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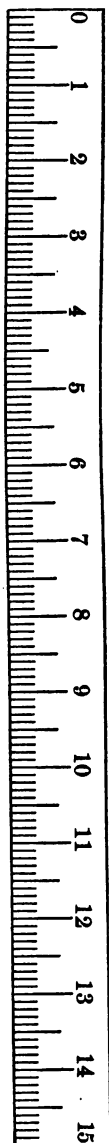
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**LABORATORY MANUAL**  
**ARRANGED TO ACCOMPANY**  
**"A COURSE IN GENERAL CHEMISTRY"**  
**SECOND EDITION**  
**McPHERSON AND HENDERSON**



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centimeter (cc.)  
decimeter = 1 liter (l.)

1000 cubic centimeters = 1 cubic meter

The chemist uses only the cubic centimeter and the liter as measures of volume. Thus, the volume of a test tube is given as (say) 25 cc.; that of a flask as (say) 500 cc. or 0.5 liter.

**3. Weight.** The unit is the *gram*. This is the one thousandth part of the weight of a piece of platinum preserved at Sèvres, France. It is equal to 15.43 grains.

10 milligrams (mg.)	= 1 centigram (cg.)
10 centigrams	= 1 decigram (dg.)
10 decigrams	= 1 gram (g.)
1000 grams	= 1 kilogram (kg.)

The gram is practically the only unit used by the chemist. Thus, the weight of a crucible is given as (say) 10.532 g. and not 10,532 mg. or 10 g. 5 dg. 3 cg. 2 mg.

Note that

- 1 pound troy = 5760 grains = 373.24 grams
- 1 pound avoirdupois = 7000 grains = 453.59 grams
- 1 U.S. quart = 0.95 liter

Also note that

- 1 centimeter = nearly  $\frac{1}{2}$  inch
- 1 kilogram = nearly  $2\frac{1}{2}$  pounds (avoirdupois)

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# LABORATORY MANUAL

ARRANGED TO ACCOMPANY THE  
SECOND EDITION OF

"A COURSE IN GENERAL CHEMISTRY"

BY

WILLIAM McPHERSON

AND

WILLIAM EDWARDS HENDERSON

PROFESSORS OF CHEMISTRY, OHIO STATE UNIVERSITY



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## PREFACE

From an educational point of view, chemistry is really the oldest of the experimental sciences. The problem as to what laboratory work should constitute the first year's course is therefore one to which a great deal of thought has been devoted, and many educational experiments have been made in an endeavor to solve it. Much ingenuity has been exercised in the development of suitable laboratory experiments and in the invention of simple apparatus adapted to a beginner's inexperience, and a great wealth of admirable illustrative experiments is now at the command of every teacher.

For one who sets about the task of arranging an experimental course for the beginner, there remains little opportunity for originality or invention. His problem is rather one of selection. Accordingly this laboratory manual lays no claim to originality, either in method or in content. It has been slowly developed in connection with the large beginning classes in which the authors have been interested, and has been revised and reprinted privately a number of times. In response to many requests it has again undergone a thorough revision and has been arranged to accompany the text by the authors, entitled, "A Course in General Chemistry."

In common with nearly all teachers of chemistry, the authors have had to deal with the fact that the first course in college comprises students who have had an elementary course in the high school, as well as those who have had no earlier introduction to chemistry. As far as the laboratory is concerned, the authors have found that the most practical solution of this problem is to develop a manual ample enough to meet the needs of both classes of students. The



more experienced student can then omit the exercises with which he is familiar, and the student with less experience can omit some of the quantitative exercises.

Every well-ordered laboratory has its own system of supplies, stock solutions, desk reagents, and locker equipment, and the directions in a manual will not always harmonize with this system. For example, in many cases in which the student is directed to prepare solutions or mixtures for specific purposes, it may be much better to have a supply on the side shelf, properly labeled, for general use. In a number of experiments, particularly those involving apparatus that is not a part of the locker equipment, two or even more students may work together to advantage. The time at the disposal of the class will not always permit each student to complete all of the exercises, and variety is added by assigning parallel experiments to alternate students. All such adjustments are left to the instructor.

In the Appendix will be found a number of suggestions to the instructor, relating to the details of special apparatus or reagents. Suggestions are also offered as to a suitable locker equipment and as to the apparatus that the student will need from time to time. Articles may obviously be transferred from one list to another according to the funds available or the capacity of the lockers. A list of the reagents called for in the manual is provided, together with an estimate of the quantities required for a class of ten students.

A large page has been chosen for the manual, since it makes a convenient book for filing in laboratory pigeonholes and for handling during correction. The blank pages can be used for a full record of laboratory observations, or more elaborate reports may be written from the recorded notes if the instructor so desires.

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## PREFACE TO SECOND EDITION

The publication of a second edition of the authors' "A Course in General Chemistry" has made it necessary likewise to revise the Laboratory Manual arranged to accompany the text. Attention is called especially to the fact that it is not expected that any one student will perform all the experiments included in the manual. Each instructor will select such as best meet the needs of his class.

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**LABORATORY MANUAL**  
**ARRANGED TO ACCOMPANY**  
**"A COURSE IN GENERAL CHEMISTRY"**

# LABORATORY MANUAL

## CHAPTER I

### MANIPULATION AND FUNDAMENTAL PRINCIPLES

1. **The Bunsen burner.** *a.* The Bunsen burner is the common form of apparatus used in laboratory operations where heat is applied. It consists of the tube *A* (Fig. 1), screwed into the base *C*. The tube has two small round holes near its lower part. A band *B*, provided with similar holes, fits around the lower part of the tube in such a way that the holes in the tube may be closed or opened by turning the band. Gas is admitted to the burner through *D* by means of rubber tubing. Unscrew the tube and examine the different parts of the burner; then put them together again and light the gas by holding a lighted match 4 or 5 cm. above the tube and turning on the gas. The gas should be adjusted so as to give a flame about 10 cm. high. The gas entering the burner mixes with air drawn in through the holes in the lower part of the tube and burns with an almost nonluminous flame. If the band is adjusted so as to close the openings, the flame becomes luminous. Always use the nonluminous flame unless otherwise directed.

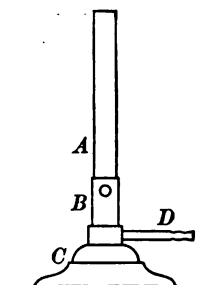


FIG. 1

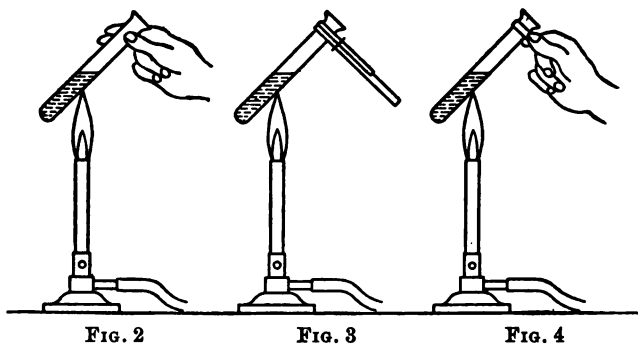
*b.* Hold an iron wire in various parts of the flame to gain an idea as to the relative temperatures. Pass a piece of glazed paper transversely through the flame, holding it steady for an instant across the flame. What information do you



get from the way in which the paper is scorched? Pass a piece of paper edgewise through the axis of the flame in the same way.

c. From these experiments draw a diagram of the flame, indicating the hot and the cooler regions.

**2. Heating a liquid in a test tube.** Hold the test tube between the thumb and fingers (Fig. 2), constantly rotating it backward and forward so as to apply the heat uniformly. The heat should be applied to the upper portion of the liquid, care being taken, however, that the flame does not strike the tube above the level of the liquid. In case the upper part of



the tube becomes heated it may be supported by a test-tube holder (Fig. 3) or by a band of paper circling the upper part of the tube (Fig. 4).

The sudden formation of vapor at the bottom of the tube sometimes causes the contents of the tube to be thrown out; hence care must be taken not to point the tube toward anyone.

Half fill a test tube with water and apply heat until the water boils rapidly.

**3. Pouring a liquid from one vessel to another.** In this operation care must be taken to prevent the liquid from running down the side of the vessel from which it is poured. A glass rod should be held lightly against the rim of the vessel, as shown in Fig. 7. The liquid will flow down the rod. Fill a beaker with water and transfer it slowly to another





vessel without using the glass rod ; repeat, using the glass rod. What difference is noted ?

In pouring a liquid from a bottle a glass rod may be used ; or the neck of the bottle may be placed lightly against the rim of the vessel into which the liquid is being poured (Fig. 5). This will prevent the liquid from running down the side of the bottle. Obviously the stopper should never be laid down on the desk. It should be caught between the fingers, as shown in Fig. 6. This leaves the hand free to grasp the bottle, as shown in Fig. 5.

**4. Decantation.** It is often necessary to separate a liquid from a finely divided solid which is suspended in it. This

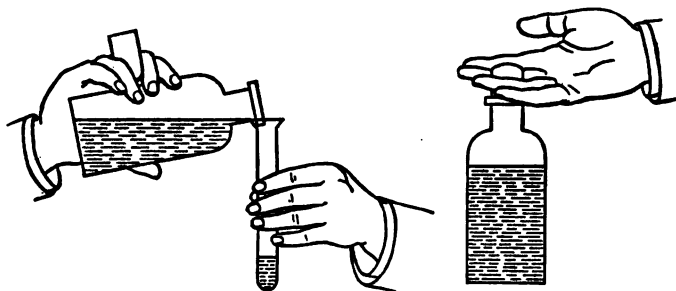


FIG. 5

FIG. 6

may be done most simply by *decantation*. The operation consists in allowing the solid to settle and then pouring off the liquid. The method can be used only when the solid is heavy and readily settles in the liquid. Shake up some fine sand in water, then allow it to settle and *decant* the water.

**5. Filtration.** As a rule the solid will not readily settle or will do so only after long standing. In such cases the mixture is *filtered* ; that is, poured on a filter paper, which allows the liquid to run through but retains the solid. To prepare the filter paper, fold it along a diameter into halves, then, at right angles to the first fold, into quarters. The folded filter is then opened so as to form a cone, half of which is composed of three thicknesses of paper and the remainder of one thickness. Fit the cone into a funnel, of such a size that the paper does



not quite reach the top. The paper must accurately fit the funnel; if it does not, make it do so by varying the fold. Place the paper in the funnel and thoroughly wet it with water. After the water has run through, press the paper firmly against the sides of the funnel with the finger so as to remove any air bubbles between the paper and the glass. The filter is now ready for use.

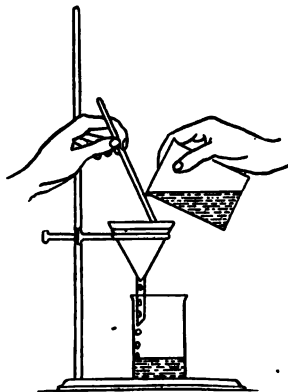


FIG. 7

The process of filtration not only enables us to separate liquids from solids but also certain solids from each other. To illustrate this fact, grind a piece of chalk to a powder in a mortar and mix the product intimately with about an equal bulk of common salt. Now separate the two solids as follows: Place the mixture in a small beaker, add about 50 cc. of distilled water, and stir with a glass rod. (The sharp ends of the rod must be rounded by rotating them in a flame, otherwise the beaker may be scratched and broken.) The salt dissolves, forming a *solution*. Filter off the insoluble chalk, collecting the *filtrate* (the clear liquid which passes through the filter paper) in a beaker (Fig. 7). The salt may be recovered from the filtrate by the process of *evaporation*. To perform this operation, pour the filtrate into an evaporating-dish, then support the dish on a ring stand (Fig. 8) and heat gently. The tip of the flame should not quite touch the dish. The liquid may be made to simmer, but should not be made to boil violently (why?). Withdraw the heat as soon as the water is evaporated. Note the residue left. Convince yourself that it is salt.

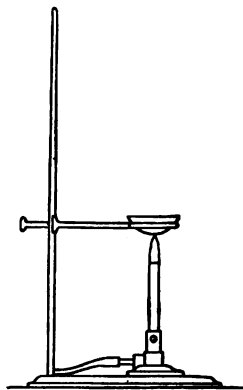


FIG. 8



**6. The metric system.** *a. Length.* By means of the scale on the front cover of the notebook measure the length (in centimeters) of various pieces of apparatus included in your outfit, as a test tube, file, and blowpipe. What is the diameter of your filter paper? Estimate the lengths of various objects, as a pencil, a test tube; then measure. Continue until you can approximate the lengths of small objects.

*b. Volume.* By means of a graduated test tube or cylinder measure (in cubic centimeters) the volumes of various test tubes, beakers, and flasks included in your outfit. (In reading the volume of the liquid in a graduated tube always read from the lower part of the *meniscus*; that is, the curved surface of the liquid.)

**7. The balance.** The weight of a body is determined on a balance, which must be adapted to the degree of precision required. For the experiments undertaken in this course the balance must weigh accurately to one one-hundredth of a gram, and, if possible, to one one-thousandth. It is convenient to remember that a gram is approximately one thirtieth of an ounce.

To be of service for even moderately accurate weighing, a balance must be used with the greatest care. The following directions must be faithfully observed:

1. At all times, save during an actual weighing, the beam must be lifted from its bearings by turning the thumbscrew at the base of the case.

2. All changing of objects to be weighed and of weights must be done after the beam has been lifted. Never take off a weight while the beam is swinging.

3. Never handle the weights in any manner save with the pliers provided with the weights. Be careful not to dent the weights by rough use with the pliers.

4. Keep the balance pans, the case, and the balance shelf scrupulously clean.

5. Never weigh chemicals directly on the pan of the balance. First weigh a suitable container,—a watch glass or a small beaker,—then place the material in this and again weigh.



**8. The process of weighing.** *a.* See that upon careful lowering of the beam the vibrations of the pointer are equal on both sides of the zero point. If there is any considerable deviation, have the instructor adjust the balance.

*b.* Place the object to be weighed on the left pan and the weights you judge to be sufficient on the right pan. Turn the arrest screw very slowly until you see in which direction the pointer starts to move. Arrest the beam and make a new adjustment of weights, repeating the operation until the desired equilibrium point is found.

*c.* Add up the weights lying on the pan, and verify the count by the empty spaces in the box. Always have the weights either on the pan or in the box, and never lay them on the floor of the balance case.

*d.* Record your weighings directly in your notebook — never on scraps of loose paper. Observe that the weights are in decimals and that their sum should be expressed in grams and decimal fractions thereof.

*e.* Select a clean watch crystal or a small beaker which you will be using for weighing. Carefully weigh it and record the weight in your notebook. With a sharp file make a slight scratch upon the beaker just below the lip. Fill the beaker with distilled water from a graduated measure and note the volume added. Weigh the beaker and water and determine the weight of the water. How does the weight in grams compare with the volume in cubic centimeters? Why is this? Make a mark on a test tube about 2 cm. from the top. Weigh the empty tube and also the tube filled with water to the mark. Determine the volume to the mark. Make use of the tube in measuring out liquids until you grow accustomed to estimating volumes in cubic centimeters.

**9. Manipulation of glass tubing.** *a. To cut glass tubing.* Place the tubing on the desk and draw the edge of a triangular file across the point at which you wish to cut the glass.

NOTE. In all operations requiring the application of a strong heat to glass the heat must be applied gently at first; also, the highly heated glass must be cooled slowly to anneal it properly.





After the glass is scratched, take the tube in the hands with the thumbs placed near together, just back of the scratch (Fig. 9), and gently pull the glass apart, at the same time exerting a slight pressure with the thumbs. Do not try to break the tubing as you would break a stick, for this will

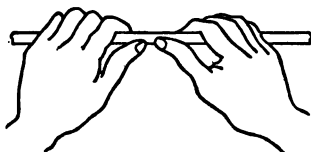


FIG. 9

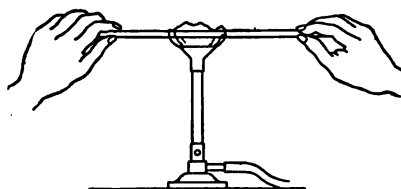


FIG. 10

splinter the glass and will never give a square end. If the tube does not yield readily to the gentle pressure, a deeper scratch must be made. In the case of large tubing it may be found necessary to file a groove around the tube. The edges of the cut tube will be sharp. They should be rounded by rotating them in the tip of the Bunsen flame. This process is called *fire-polishing*.

*b. To bend glass tubing.* Use the luminous Bunsen flame, spread out by means of the so-called "wing-top" burner (Fig. 10). Hold the tube lengthwise in the flame, gently rotating it so that all sides may be equally heated. Continue the heating until the glass *easily* bends, then quickly remove it from the flame and bend to the desired shape (Fig. 11, *A*). Great care must be taken to heat the tube uniformly, otherwise the bore of the tube will be contracted (Fig. 11, *B*), forming a bend which not only is unsightly but is easily broken.

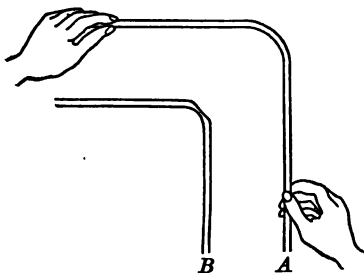


FIG. 11

*c. To insert a glass tube in a cork.* The cork should be of such a size that the smaller end will just enter the flask or



bottle in which it is to be used. Soften the cork by rolling it between the desk and a piece of wood. To insert a glass tube select a borer slightly smaller than the tube. Place the cork on the desk and cut about halfway through it, not by punching, but by rotating the borer under gentle pressure (Fig. 12). Reverse the position of the cork and cut through from the other end. Care must be taken to keep the borer at right angles to the cork. The hole should be straight and smooth. The glass tube, rounded at the edges, is now inserted by a gentle screwlike motion. If the hole is too small to admit the tube when a gentle pressure is applied, it may be slightly enlarged with a round file. The tube slips in better if both cork and tube are wet.

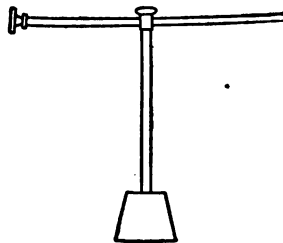


FIG. 12

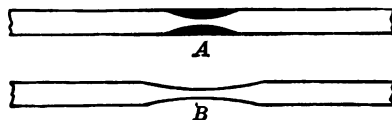


FIG. 13

*d. To draw out a glass tube to a small bore.* Heat a portion of the tube in the Bunsen flame until the walls of the heated portion thicken and the size of the bore diminishes (Fig. 13, A). The tube must be constantly rotated to prevent the softened portion from sagging. Now quickly remove the tube from the flame, and, holding it in a vertical position, gently pull the ends apart until the bore is of the desired size (Fig. 13, B). A glass jet may be formed by cutting the tube at B, and rounding the edges in a flame.

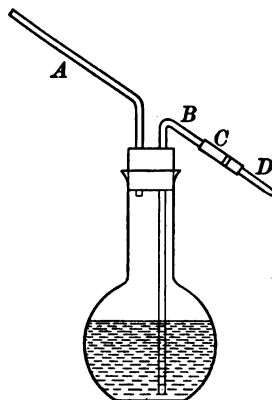


FIG. 14

*e.* After practicing the above manipulations, make a wash bottle according to Fig. 14. Use a 500-cc. flask. A and B are glass tubes. B is connected with the



glass jet *D* by a piece of rubber tubing *C*. The edges of the glass tubing must be rounded. When the wash bottle has been approved by the instructor, fill it with distilled water and set it aside for future use.

**10. Chemical action.** *a.* Hold a piece of iron wire in the Bunsen flame for a few seconds. Is the iron changed? Examine it when it has cooled. Have the properties changed? Has a chemical action occurred?

*b.* Repeat *a*, using a splint of wood in place of the iron wire. How does the change produced differ from that in *a*? Has a chemical action occurred?

*c.* Place enough sugar in a clean, dry test tube to cover the bottom to a depth of 1 cm. Heat it gently in the tip of the flame as long as any changes are produced. Note all the changes. Is the product sweet? Is it soluble in water? Do any properties remain unchanged? What grounds do you have for assuming that a chemical action has taken place?

*d.* Place about 1 g. of common salt in a test tube and dissolve it in a little water. Pour the clear solution into an evaporating-dish and evaporate to dryness. What is the solid left? How do its properties correspond to those of the original salt?

*e.* Cover a small piece of zinc in a test tube with about 5 cc. of water and add carefully 3 or 4 drops of sulfuric acid. Notice that the zinc dissolves with the evolution of a gas. Hold a burning splint at the mouth of the test tube and note the result. After the action has entirely ceased, filter off any undissolved zinc and evaporate the solution to dryness (hood) as in *d*. How does the change differ from that in *d*? Distinguish between the two examples of solution.

**11. Elements; compounds; mixtures.** *a.* What is an element? Are iron and sulfur included in the list of elements? Weigh out separately (on paper) 2 g. of sulfur and 2 g. of clean iron filings and make a careful list of their properties. Try the effect of a magnet on each. Now mix the two, and grind them together intimately in a mortar. Examine the product with a magnifying-glass. Can you distinguish the



iron from the sulfur? Can you separate them with a magnet? Have they undergone any change in properties? What is such a material called?

**b.** Now place the product in a clean test tube and heat gently. As soon as the mass begins to glow, quickly withdraw the tube from the flame. Does the mass continue to glow? Now heat it strongly for one or two minutes; then cool the tube, break it, and examine the product with a magnifying-glass. Can you now distinguish between the iron and the sulfur? Try the effect of the magnet. Of what is the substance composed? When elements combine chemically, do they retain their original properties? What is the product of such a combination called?





## CHAPTER II

### OXYGEN

**12. Collection of gases.** Fill a wide-mouthed bottle (250-cc.) with water. Cover its mouth with a glass plate, being careful to exclude all air bubbles. Hold the plate firmly in place, invert the bottle, and bring its mouth below the surface of the water contained in a suitable vessel. The most convenient vessel is a rectangular box of sheet iron called a *pneumatic trough* (Fig. 16). Remove the glass plate. Why does the water remain in the bottle? Now fill the bottle with exhaled air by placing one end of a piece of glass or rubber tubing under the mouth of the bottle and blowing gently through the other end. The bubbles will rise and fill the bottle.

Before the bottle, so filled, is removed from the trough, cover its mouth tightly with a glass plate. The bottle so covered may then be placed on the desk either right side up or in an inverted position. (When should it be placed in an inverted position?)

Fill a bottle with exhaled air and transfer the air so collected to another bottle. Draw a diagram to illustrate a suitable method of doing this.

**13. Preparation of oxygen.** *a.* In the bottom of a clean, dry test tube place about 0.5 g. of mercuric oxide. This is best accomplished by placing the oxide near the end of a narrow strip of folded paper and introducing it carefully into the tube, as shown in Fig. 15. On inclining the tube and gently tapping the paper, the oxide will be deposited in the bottom of the tube. The paper is then withdrawn, leaving



FIG. 15



the sides of the tube perfectly clean. Hold the tube between the thumb and fingers (Fig. 2) and apply a gentle heat to the oxide. The tube must be rotated constantly to distribute the heat; otherwise the glass may soften. While the heating is maintained, insert a glowing splint from time to time into the mouth of the tube. Note the result. Continue to heat as long as any gas is evolved. What remains in the tube? How has the heat affected the mercuric oxide?

*b.* In a similar way try heating other oxides such as iron oxide, manganese dioxide, lead peroxide, barium dioxide, and copper oxide. Do all of these yield oxygen?

**14. Catalysis.** Place about 1 g. of potassium chlorate in a test tube and carefully heat it in a small Bunsen flame until it has just melted. Can you see any gas being given off? Continue the heating until you can plainly see that gas is liberated. What is it? From the time of heating would you suppose that the temperature had been raised much above the melting point? Allow the liquid to cool until it begins to solidify, and then just melt it again. Then add a very little powdered manganese dioxide, dropping it in from a small knife blade. What effect is produced? In preparing oxygen from the chlorate what would be the advantage of adding manganese dioxide before heating? In place of manganese dioxide try the effect of iron oxide and of powdered sand.

**15. Usual laboratory method.** Arrange an apparatus according to Fig 16, in which *B* is a hard-glass test tube, *D* a glass tube bent at right angles, and *C* a piece of rubber tubing. Mix intimately on paper 6 g. of potassium chlorate and 3 g. of manganese dioxide. Transfer the mixture to a hard-glass tube and insert the cork; then, holding the burner in the hand, heat the mixture gently with a small flame, applying the heat at first to the upper part of the mixture. The flame must not strike the upper part of the test tube, as the cork may be ignited. At first the heat expands the air and a few bubbles escape; at a higher temperature the oxygen is evolved. Regulate the heat so as to secure a uniform and not too rapid evolution of the gas. By displacement of water collect three

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or four wide-mouthed bottles (250-cc.) of the gas. Before the heat is withdrawn remove the cork from the tube (why?).

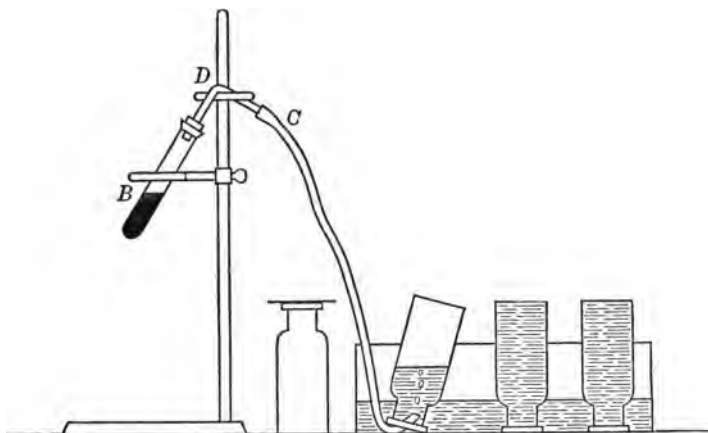


FIG. 16

Reserve the tube with its contents for study in § 19. What is the source of the oxygen? What is the function of the manganese dioxide?

**16. Preparation from sodium peroxide and water.** Sodium peroxide is a white solid containing 41 per cent of oxygen, and when treated with water, a part of this is set free. Arrange an apparatus according to Fig. 17. By means of a short piece of rubber tubing *A* connect the funnel *B* with a glass

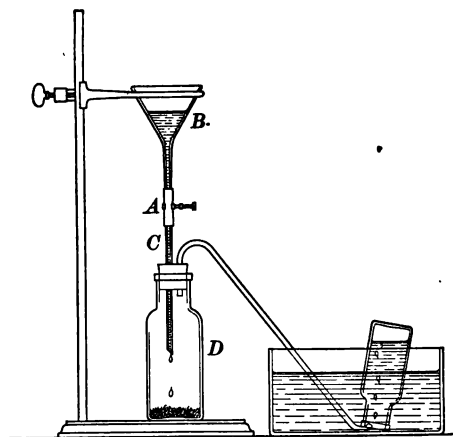


FIG. 17

tube *C*, pinching the tube shut with a screw clamp. Place about 5 g. of sodium peroxide in the bottom of *D* and partly



fill the funnel with warm water. Very cautiously open the screw clamp so that the water will run down and fall, drop by drop, upon the peroxide. A steady current of oxygen is given off, and the gas should be collected as in the preceding exercise. (A separatory funnel may be used to advantage instead of the funnel and screw clamp.)

**17. Preparation from potassium permanganate and hydrogen peroxide.** Using the apparatus shown in Fig. 17, place a solution of 5 g. of potassium permanganate in 25 cc. of water in *D* and add 3 cc. of sulfuric acid. Fill the funnel with commercial hydrogen peroxide and allow it to flow very slowly into the solution in *D*.

**18. Properties and conduct of oxygen.** *a.* Note the properties of the gas. (The slight cloud often present when oxygen is prepared from potassium chlorate is due to an impurity and will disappear if the gas is allowed to stand over water.)

*b.* Repeatedly thrust a glowing splint into a bottle of the gas.

*c.* Heat some sulfur in a deflagrating-spoon until ignited. Note the color and size of the sulfur flame. Now lower the burning sulfur into a bottle of oxygen and note the change.

*d.* Tip a piece of picture-frame wire, 12 or 15 cm. long, with sulfur by wrapping a bit of cotton about the end of the wire and dipping this into melted sulfur. Ignite the sulfur by holding it in a Bunsen flame for an instant, then thrust the wire into a bottle of oxygen.

Are the changes observed in *b*, *c*, and *d* examples of chemical action? What becomes of the oxygen? What is the name of the product formed in each case?

**19. Separation of the compounds present in the residue left in the preparation of oxygen.** Heat the tube containing the residue obtained in § 15 until no more oxygen is evolved. After the tube is cool, half fill it with water and shake the contents thoroughly. After a few minutes filter off the solid matter (what is it?). Evaporate the filtrate to a volume of 4 or 5 cc., and set it aside until crystals are deposited. Convince yourself that the substance is different from the potassium chlorate with which you started.





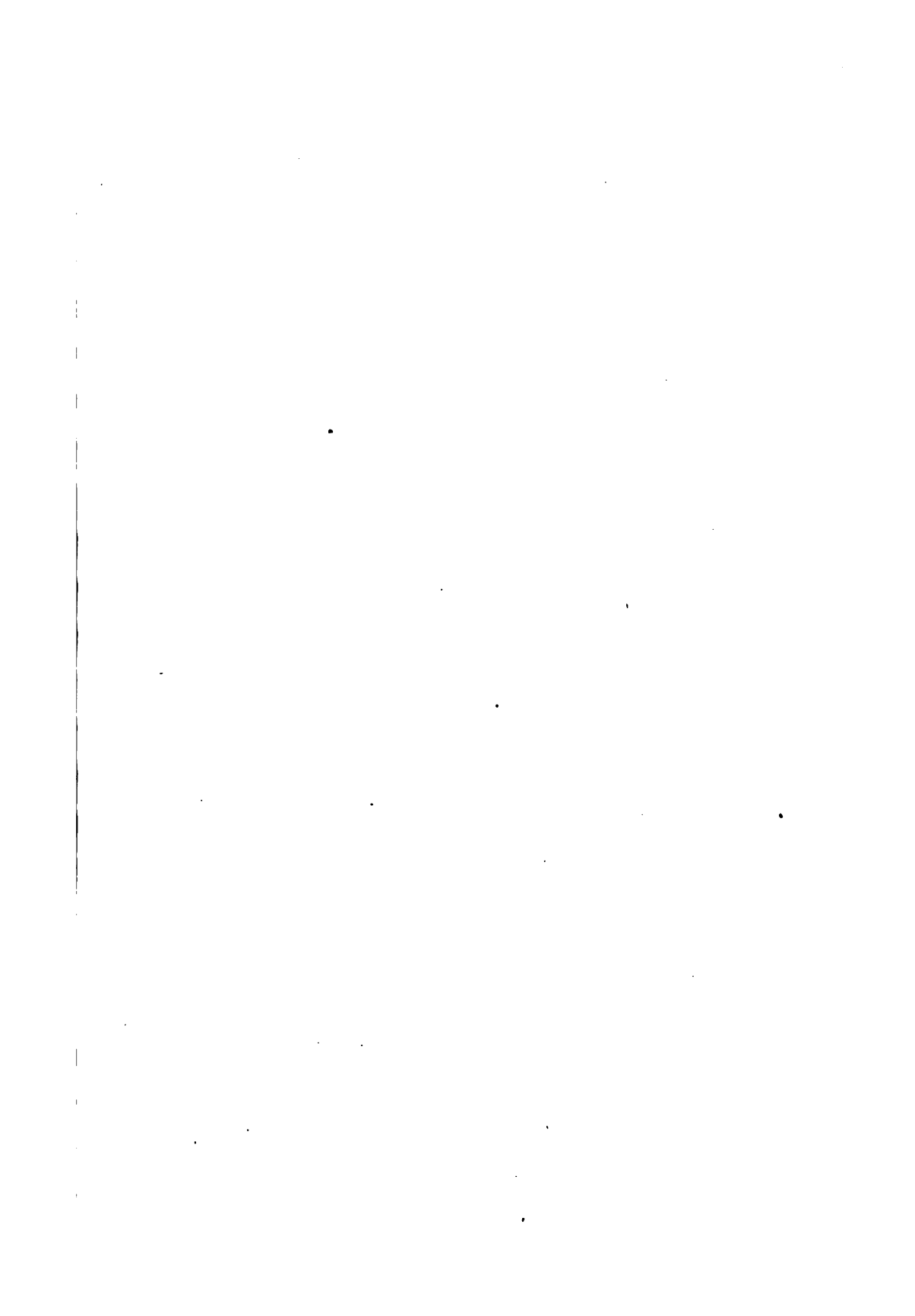
## CHAPTER III

### HYDROGEN

**20. Preparation of hydrogen from water.** Fill a test tube with water and invert it in a beaker of water. Wrap a piece of sodium, the size of a small pea, in a bit of filter paper previously moistened with coal oil. Raise the inverted test tube until its mouth dips just below the surface of the water in the beaker, and quickly insert the sodium. Stand at arm's length, as a slight explosion sometimes occurs. Notice that the sodium decomposes the water, liberating a gas which is caught in the tube. Holding the tube mouth downward, test the gas by quickly inserting a burning splint. Does the gas act like oxygen? What is the source of it? What other methods may be employed for obtaining it from the same source?

**21. Preparation from sodium hydroxide.** Provide a hard-glass test tube with a stopper and a delivery tube, as shown in Fig. 16. Finely powder about 2 g. of solid sodium hydroxide in a mortar and mix it with a little more than an equal weight of zinc dust. Place the mixture in the bottom of the test tube, clamping the tube in a nearly horizontal position, the bottom being slightly higher than the mouth (why?). Heat the mixture carefully, collecting the gas evolved in a test tube by displacement of water. Test it with a lighted splint as in § 20. What is the source of the gas?

**22. Preparation from acids.** *a.* In clean test tubes place samples of the following metals: iron, tin, lead, magnesium, aluminium, zinc, and copper. Prepare some dilute hydrochloric acid by adding about 20 cc. of the concentrated reagent to an equal volume of water. Pour 4 or 5 cc. of the dilute acid in each of the test tubes, gently warming the solution



if no action is noticed. In which cases do you observe the formation of gas bubbles? Where do they form? Do not mistake bubbles of air or steam for the gas. How can you tell the difference? Does the condition of the metal make any difference (compare the action of the acid on a tack with its action on iron filings)? Do all of the metals yield hydrogen with acids?

*b.* Repeat the experiment, replacing the hydrochloric acid with nitric acid diluted with an equal volume of water. In what cases is a gas evolved? What differences do you note as compared with *a*? In each case test the gas with a lighted

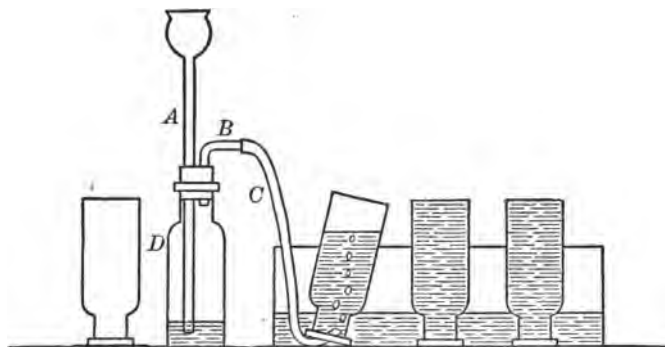
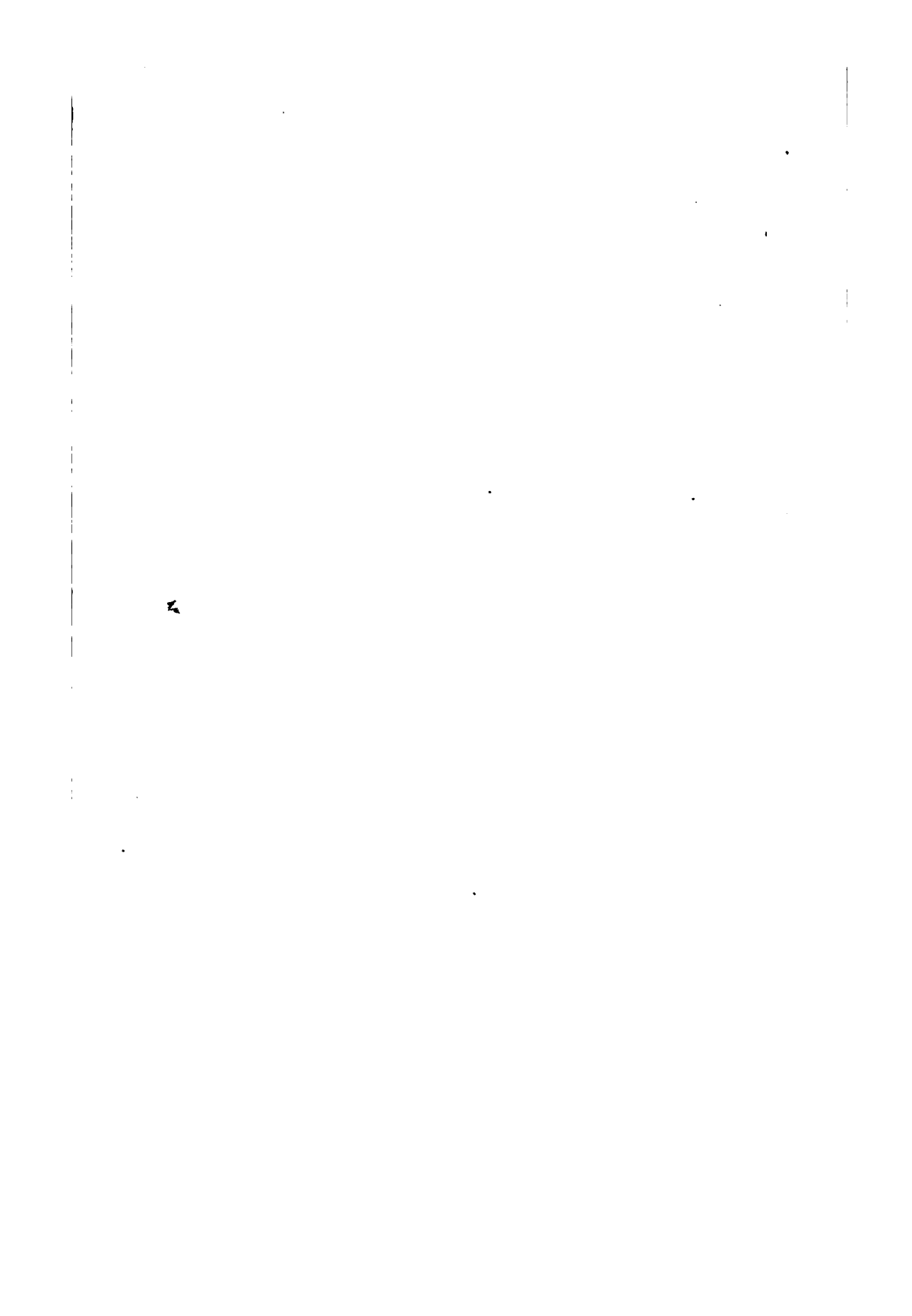


FIG. 18

splint. Is it hydrogen? Do the metals which give hydrogen with hydrochloric acid give it with all acids?

*c.* Select two test tubes and in each place a few pieces of mossy zinc, adding enough water to cover the metal. To one test tube add a few drops of a solution of copper sulfate. What change do you notice? Now add to each about 1 cc. of hydrochloric acid. Which evolves hydrogen the more rapidly? What is the function of the copper sulfate (§ 14)?

**23. Usual laboratory method.** Arrange a hydrogen generator according to Fig. 18, in which *D* represents a wide-mouthed bottle of about 250-cc. capacity. The gas delivery tube *B*, *C* is the same as that used in the preparation of oxygen (Fig. 16). The funnel tube *A* must extend nearly to the bottom of the bottle (why?). Put 10 g. of mossy zinc (why mossy



zinc?) into *D* and add a few drops of a solution of copper sulfate. Pour just enough water through the funnel tube to cover the zinc. Prove that the apparatus is air-tight by blowing into the delivery tube until the water is forced nearly to the top of the funnel tube; then quickly close the rubber tube either by tightly pinching it or by placing the tongue firmly against its end. If the apparatus is air-tight, the water in the funnel tube will not fall (why?). Prepare some dilute sulfuric acid by *slowly* pouring 15 cc. of concentrated acid into a beaker containing 50 cc. of water. Stir the water with a glass rod while the acid is being added. *Notice that the acid is poured into the water — never the reverse.* Cool the mixture and pour a few drops of it through the funnel tube. Hydrogen is at once evolved. Enough of the acid must be added from time to time to cause a gentle and continuous evolution of the gas. It is evident that the first gas which passes over is a mixture of hydrogen and air. *The student must remember that such a confined mixture of hydrogen and air or hydrogen and oxygen, if ignited, explodes with great violence.* On this account see that the end of the delivery tube is not brought near any flame. Determine when the hydrogen is free from air by collecting test tubes full of gas and igniting it, holding the tube mouth downward. If pure, the gas burns quietly, otherwise there is a slight explosion. After all the air has been expelled from the generator, collect four bottles (250-cc., wide-mouthed) of the gas. What is the source of the hydrogen? What is the use of the zinc? Why is the copper sulfate solution added? Remove the stopper from the generator, add a few more fragments of zinc, and set the generator aside until the gas ceases to be evolved. Sufficient zinc should be used so that at least a small portion of it remains undissolved. Filter the liquid from the undissolved zinc into an evaporating-dish and evaporate it to dryness. What is left in the dish? After the dish is cool, dissolve the residue in as little hot water as possible and set it aside until crystals are deposited. Compare these with crystals of zinc sulfate.



**24. Properties of hydrogen.** *a.* Thrust a lighted splint into a bottle of the gas held mouth downward. Slowly withdraw the splint and again thrust it into the gas. Describe the results. What do they prove?

*b.* Fill a small (60-cc.) wide-mouthed bottle or a test tube one third full of water, and invert it in a pneumatic trough. Displace the remaining water with hydrogen. What does the bottle now contain? Withdraw it from the water, and, holding it at arm's length, quickly bring it mouth downward over a flame. What do the results prove?

*c.* Uncover a bottle (mouth upward) of the gas. After one minute test for the presence of hydrogen with a lighted splint. Repeat, keeping the bottle mouth downward. Describe the results. Is the gas heavier or lighter than air?

*d.* Wash out the hydrogen generator, then remove the rubber delivery tube and connect the tube *A* (Fig. 19) with the drying-tube *B* by a short piece of rubber tubing. The tube *B* is filled with pieces of calcium chloride (why?), held in place by loose plugs of cotton at each end of the tube. The outer end of the bent glass tube *C* is drawn to a jet. After the apparatus has been approved by the instructor, charge the generator with 6 or 8 g.

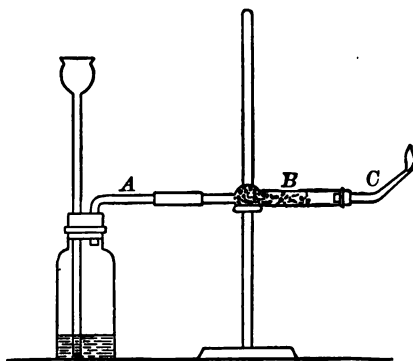


FIG. 19

of zinc, add a few drops of a solution of copper sulfate, cover the zinc with water, and add dilute sulfuric acid as in § 23. *After all the air has been expelled* (prove this by slipping a piece of rubber tubing over the tube *C* and testing samples of the gas collected over water) wrap a towel carefully about the generator and cautiously ignite the hydrogen. Test the heat of the flame by holding in it different objects, such as a splint, a piece of picture-frame wire, a bit of charcoal.





**25. The oxyhydrogen blowpipe.** If the instrument is available, examine the structure of the oxyhydrogen blowpipe. Draw a diagram representing a cross section of it. Compare it with the ordinary laboratory blowpipe (blast lamp). Why not have a short inner tube instead of a long one?

**26. Qualitative synthesis of water.** Generate hydrogen as in § 24, *d* (note precautions). Ignite the hydrogen and hold over the flame a cold, dry beaker. What substance condenses on the sides of the beaker? Account for its formation. The method of formation proves the presence of what elements in this substance?

**27. Reduction.** Arrange an apparatus according to Fig. 20, in which a hard-glass tube *C*, about 30 cm. long and from 8

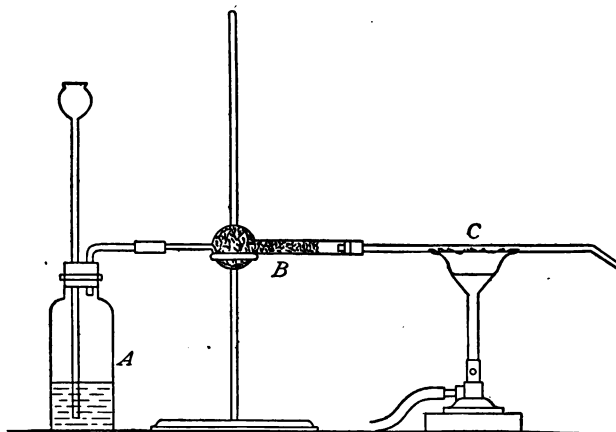


FIG. 20

to 10 mm. in diameter, is substituted for the glass jet *C* of Fig. 19. Spread 1 g. of black copper oxide in a layer in the tube 10 or 12 cm. from the end which is connected with the drying-tube. This is done as shown in Fig. 15, except that the tube must be held in a horizontal position and the oxide deposited at the proper place by turning the paper over. Now generate hydrogen as in § 23. After all the air has been expelled from the apparatus and the generator has been wrapped in a towel, cautiously heat the copper oxide to



redness with a "wing-top" burner. Note the condensation of moisture in the cold portions of the tube. Account for its formation. What change has the copper oxide undergone? Is there any visible evidence of this change? Suggest a method of finding the weight of the water formed.

**28. Oxidation.** *a.* Without removing the contents of the hard-glass tube, replace the hydrogen generator and drying-tube by the oxygen generator described in § 15. Pass a *slow* current of oxygen through the tube, gradually heating its contents to redness. What change takes place? How does the product compare with the original copper oxide?

*b.* Explain the terms *reduction*, *oxidation*, *reducing agent*, and *oxidizing agent*, and give examples of each from § 27 and § 28, *a.*

**29. Measurement of gas volumes.** *a.* Partly fill a graduated tube *A* (Fig. 21) with water and invert it in a vessel of water so that the level of the liquid in the tube is above that of the liquid in the vessel. Clamp it in this position and read the volume of the inclosed air. What conditions affect the volume of a gas? What are the conditions adopted as standard in the measurement of gases? From the volume as measured above, calculate the volume under these conditions.

*b.* Adjust the tube so that the level within is some distance below the level without. Read the new volume and reduce to standard conditions. If the liquid employed had been mercury instead of water, what changes would this make in the calculation? The correction for aqueous tension is explained on pages 91, 92 of the text and should be studied at this point.

**30. The law of Gay-Lussac (or of Charles).** The expansion of a definite volume of air through a definite interval of temperature may be measured as follows: Provide an Erlenmeyer or a Florence flask of about 200-cc. capacity, *A* (Fig. 22), and

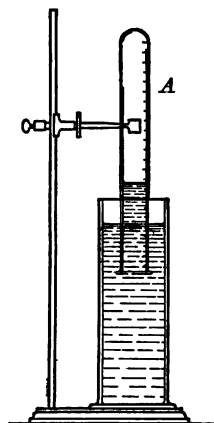


FIG. 21



fit it with a rubber stopper carrying a glass tube *B*, about 8 cm. in length and not too small in bore. When tightly inserted, the stopper should be about halfway in the flask, as shown in Fig. 22. Immerse the dry, empty flask in a beaker of water, weighting it down with a ring clamp *C*, as shown in the figure, or holding it in place by clamping the glass tube. Heat the bath at the boiling point of water for at least fifteen minutes, when it may be assumed that the air in the flask is also at that temperature ( $T_2$ ).

Press a finger tip firmly against the end of the tube, quickly lift the flask from the bath, using a towel or a test-tube clamp to protect the fingers, and dip the end of the tube below the surface of water in a pneumatic trough. Now remove the finger from the end of the tube and allow water from the tap to run on the bottom of the flask until the air in the flask has cooled to the temperature of the tap water. Note this temperature ( $T_1$ ). The volume of water entering the flask is equal to the contraction in the original volume ( $V_2$ ) of air in cooling from  $T_2$  to  $T_1$ .

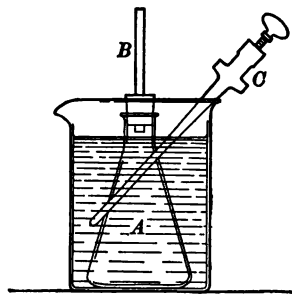
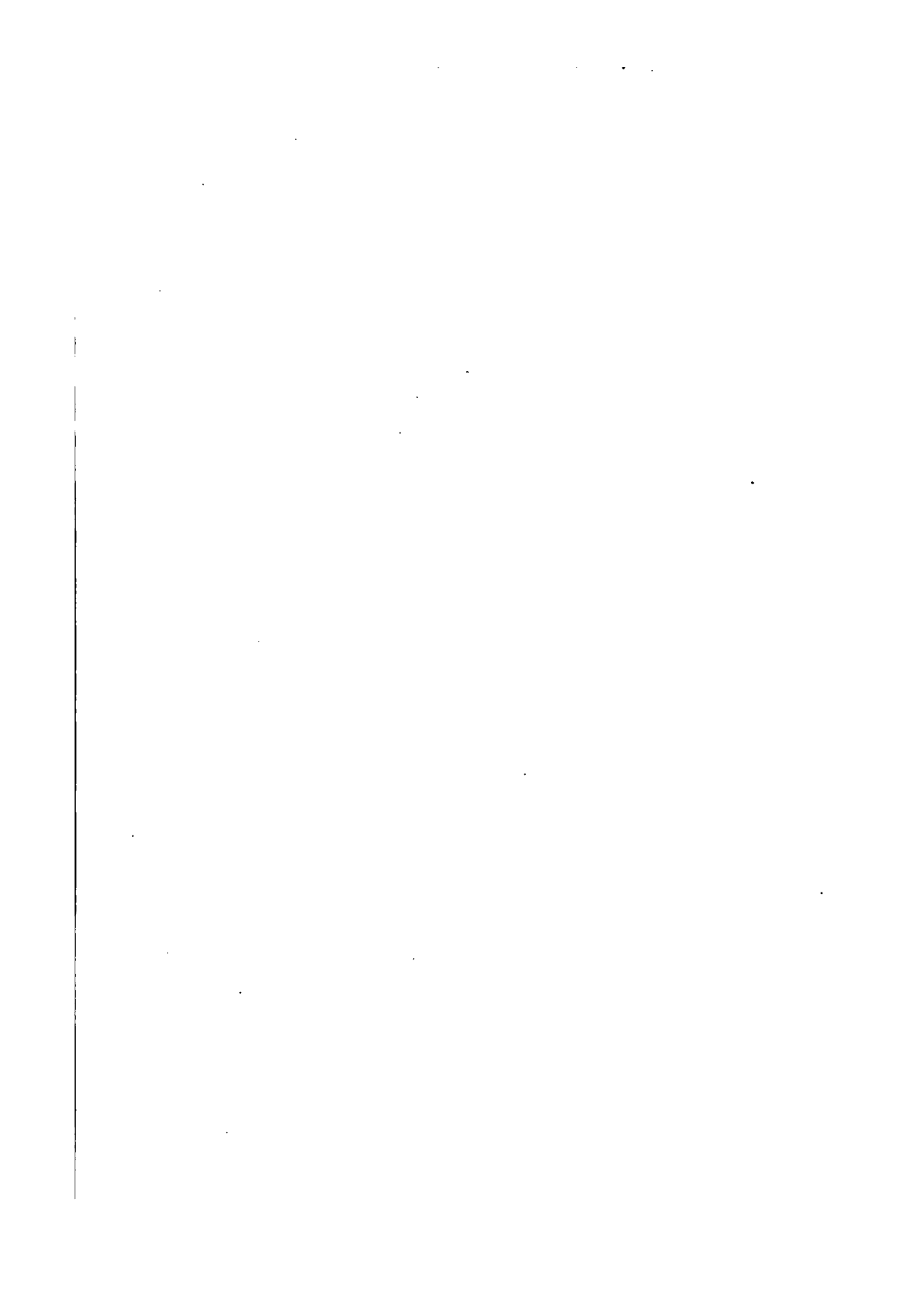


FIG. 22

Adjust the level within and without the flask, close the end of the tube, and remove the flask, placing it right side up on the table. Wipe it dry, paste a paper label to mark the level of the lower end of the stopper (or mark it with a rubber band), and cautiously remove the stopper, letting any water remaining in the tube run into the flask. Carefully pour the water into a graduated cylinder and note its volume as accurately as possible, calling this  $a$ . Fill the flask with water, pushing in the stopper to the mark and taking care that the tube is also full and that there is no air bubble under the stopper. Carefully remove the stopper, allowing the tube to drain into the space vacated by the stopper. Lastly, measure the water in a graduated tube, calling this volume  $V_2$ .



Now  $V_2 - a$  is the volume of air at  $T_1$  (volume  $V_1$ ) which will just fill the flask and tube (volume  $V_2$ ) at  $T_2$  ( $100^\circ \text{ C.}$ , or  $373^\circ \text{ A.}$ ). In other words, in the interval  $T_2 - T_1$  the gas contracts from  $V_2$  to  $V_1$ . Calculate the volume that  $V_2$  ought to give at  $T_1$ , assuming the accuracy of the law of Gay-Lussac. Compare the value found by calculation with the experimental value. What is the percentage of error? What sources of error can you think of?

**31. Percentage of oxygen in potassium chlorate; weight of 1 liter of oxygen.** Prepare the apparatus shown in Fig. 23.  $A$  represents the hard-glass test tube used in the preparation of oxygen,  $B$  is a common narrow-mouthed bottle, having a capacity of about 1 liter. The

rubber tube  $C$  is provided with a screw clamp  $D$  for closing the tube, and ends in a piece of glass tubing drawn out to a jet, the internal diameter of the jet being about 2 mm. The bottle is nearly filled with water, as shown in the figure, and allowed to stand until it acquires the room temperature. The tube  $A$  is now removed, and

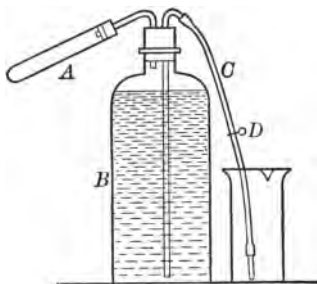


FIG. 23

a gentle suction is applied to the glass jet, at the end of tube  $C$ . The water siphons over through the tube  $C$  into the beaker and is allowed to run for a moment to fill completely both the rubber tube and the glass tube. The rubber tube is then quickly closed with the screw clamp.

Now thoroughly clean and dry the tube  $A$ , and carefully weigh it; then introduce about 1 g. of potassium chlorate into the bottom of the tube by means of a folded paper (Fig. 15), and reweigh. Attach the tube, as shown in Fig. 23, care being taken to have the apparatus air-tight. The pressure of the air within the bottle is now adjusted to that of the air outside, as follows: Water is added to the beaker (if necessary) until the end of the glass tube is covered. The screw clamp is then opened and the beaker at once raised vertically





until the water in the beaker is at the same level as the water in the bottle; then the screw clamp is closed. Empty the beaker, and return it to the position shown in Fig. 23. Now open the screw clamp and apply a gentle heat to the potassium chlorate. Oxygen is evolved and forces the water from the bottle into the beaker. Gradually increase the heat, and continue the heating until all the oxygen has been expelled. Let the apparatus stand until it has acquired room temperature, care being taken that the glass jet is kept below the surface of the water in the beaker (why?). Now bring the level of the water in the beaker to that of the water left in the bottle, and, while holding it in this position, close the screw clamp (why?). Carefully measure the water in the beaker; also take the readings of the thermometer and the barometer. Disconnect the tube *A*, and carefully reweigh the tube and its contents. Insert the values in the following table:

Weight of tube <i>A</i> . . . . .	
Weight of tube <i>A</i> + the potassium chlorate. . . . .	
Weight of tube <i>A</i> + the potassium chloride . . . . .	
Volume of water in beaker = volume of oxygen evolved . .	
Temperature of water . . . . .	
Barometric reading . . . . .	

From your results calculate (1) the percentage of oxygen in potassium chlorate, and (2) the weight of 1 liter of oxygen under standard conditions. Compare your results with those given in the text.

If sufficient heat has not been applied to expel all the oxygen from the potassium chlorate, incorrect results, of course, would be obtained for the percentage of oxygen present. This would have no effect, however, upon the determination of the weight of 1 liter of the gas. It is well, therefore, after the tube *A* and the residue have been accurately weighed, to reheat the tube and reweigh. This process should be repeated until the tube and contents suffer no loss of weight on heating.



## CHAPTER IV

### WATER AND HYDROGEN PEROXIDE

**32. Distillation.** Connect a 500-cc. flask *A* with a Liebig condenser *B* (obtained from the general storeroom), as shown in Fig. 24. The tube *C* is connected with the water pipe by means of rubber tubing, and a current of cold water is allowed to flow through the outer tube of the condenser. Why is cold water admitted at *C* rather than at *D*? Half fill the flask with hydrant water and boil till 100 cc. or more of liquid has collected in *E*, the receiver. Compare the *distillate* (distilled water) with the hydrant water in appearance and taste. To what is the difference due? Interrupt the process of distillation and add a few crystals of a highly colored salt (potassium permanganate) to the water in the distilling-flask. Continue the distillation of the water. Is the distillate colored? Place 4 or 5 drops of the distilled water on a watch glass and evaporate. Is there any residue? Repeat, using hydrant water. Why is distilled water used in the laboratory?

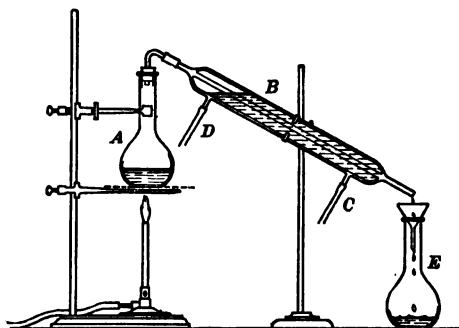


FIG. 24

tion and add a few crystals of a highly colored salt (potassium permanganate) to the water in the distilling-flask. Continue the distillation of the water. Is the distillate colored? Place 4 or 5 drops of the distilled water on a watch glass and evaporate. Is there any residue? Repeat, using hydrant water. Why is distilled water used in the laboratory?

**33. Filtration.** *a.* To about 25 cc. of water in a beaker add 1 or 2 cc. of ammonia water and stir. Note the odor of the solution. Add as much bone black or powdered charcoal as you can pile on a five-cent piece, and thoroughly stir. Filter



the solution and note the odor of the filtrate. How does it compare with that of the original solution?

**b.** To about 100 cc. of water add enough blue litmus solution to give a decided blue color. Half fill a test tube with the solution and set it aside. To the remainder add 5 cc. of a solution of alum and an equal volume of dilute ammonia. Note the formation of a white precipitate. Does it remain entirely white? Does the solution remain as blue as at first? Stir the solution for a time and pour some of it on a filter, catching the filtrate in a test tube, until the latter is half full. Compare the color of the filtrate with that of the original solution.

**34. Solid residue. a.** Nearly fill a weighed evaporating-dish with a measured volume of water (not distilled), evaporate to dryness, and reweigh. From your results calculate the weight of solid matter in a liter of water.

**b.** Accurately weigh a small evaporating-dish and a glass stirring-rod. Introduce about 10 cc. of milk and reweigh. Evaporate to dryness on a water bath (Fig. 25), stirring the contents often with the glass rod. When the liquid has entirely evaporated, remove the beaker from the bath, cool, and weigh. From your results determine the percentage of residue in the sample of milk. Vinegar may be used instead of milk.

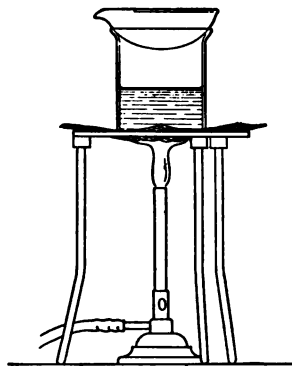


FIG. 25

**35. Chemical conduct of water. a.** What is the effect of a moderate degree of heat upon water? Under what circumstances may water be decomposed by heat?

**b. Action upon oxides.** Dip a strip of blue litmus paper into pure water. Is there any change in color? Repeat with red litmus paper. Acids turn blue litmus red, while bases turn red litmus blue. Has water either acid or basic properties? In a test tube shake a small piece of lime (the size of a pea) with about 5 cc. of water. Test the solution with litmus



paper of both colors. What inference do you draw? Set fire to a piece of sulfur supported on a deflagrating-spoon and lower it into a bottle containing a little water. After it has burned for a time, remove the spoon, cover the mouth of the bottle with the palm of the hand, and shake the bottle vigorously. Test the water with litmus paper. What conclusions do you draw? When water acts upon an oxide, what classes of substances may result?

**36. Water of crystallization, or water of hydration.** *a.* Heat some small crystals of zinc sulfate in a dry test tube. What evidence have you of the presence of water in the crystals? Examine the residue. How does it differ from the original crystals in appearance and in composition?

*b.* Select some small crystals of copper sulfate. Are they dry? Fill a test tube one fourth full of these crystals, and heat as in *a*. Compare the residue with the original crystals in form, color, and composition. Dissolve the residue in as little hot water as possible, pour the solution (note the color of it) into an evaporating-dish, and set aside until crystals are deposited. How do these compare with the original crystals of copper sulfate?

Does water seem to be necessary to the existence of zinc sulfate and copper sulfate in the usual crystalline form? What is such water called? Is it combined with the substance composing the crystal or simply mixed with it? Give reason for your answer. Do all crystals contain it? (Try a crystal of potassium dichromate.)

*c.* Expose a clear crystal of sodium sulfate to the air for one or two hours. Notice the change in its appearance. To what is the change due? What are such substances called?

*d.* Place a small piece of calcium chloride or of calcium nitrate on a watch glass and expose it to the air for two hours or longer. Note the change. What term is applied to bodies that undergo this change?

**37. Percentage of water of hydration in copper sulfate.** Accurately weigh a porcelain crucible and cover. Then add 2 or 3 g. of *small* crystals of copper sulfate and again accurately





weigh. Place the covered crucible on the pipestem triangle and heat with a gentle flame until the color of the crystals has entirely disappeared. This will require from twenty to thirty minutes. The tip of the flame must not quite touch the crucible. The product is anhydrous copper sulfate. When the crucible is cool, reweigh. From your results calculate the percentage of water of hydration in the crystals. Compare your results with those of other students who have used different weights of crystals.

**38. Quantitative synthesis of water.** Arrange an apparatus as shown in Fig. 26, in which *A* represents the hydrogen

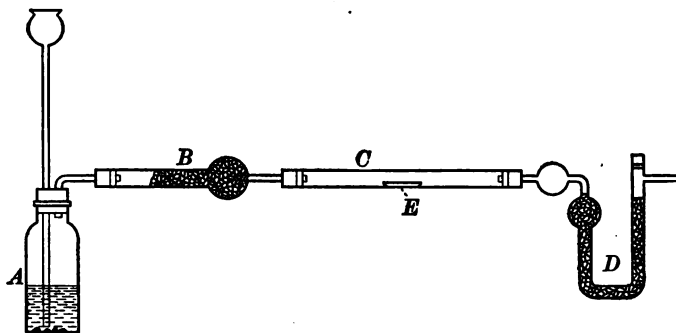


FIG. 26

generator, and *B* and *D* are tubes filled with dry calcium chloride. The hard-glass tube *C* and the porcelain boat *E* are obtained from the storeroom. The tube is about 35 cm. in length. Introduce about 2 g. of black oxide of copper into the boat, and weigh accurately to milligrams. Introduce the boat into the glass tube so that the end of the boat is about 8 cm. from the end of the tube connected with *D*. Close the ends of tube *D* with short pieces of rubber tubing, one end of each being closed with a small glass rod; then weigh the tube accurately. Remove the rubber tubes and glass rods, carefully preserving them for use when the tube is again weighed. Now connect the apparatus as shown in the figure, taking care to render it air-tight. How is this determined? Generate hydrogen slowly, and when the apparatus is free



from air, heat the boat very gently, using the wing-top burner. Gradually increase the heat, all the time maintaining the slow current of hydrogen. When the copper oxide is reduced, or nearly so, withdraw the heat but maintain the current of hydrogen until the apparatus is cool. If any of the water formed remains condensed in the end of tube *C*, a very gentle heat is cautiously applied (the flame must not strike the tube) until it is driven into the tube *D*.

When the apparatus has acquired the room temperature, disconnect *A* and attach *D* (Fig. 26) to the short bent glass tube in bottle *B* of Fig. 23. The bottle is filled with water, and a portion of it is slowly siphoned over through *C*, *D* (Fig. 23). In this way a current of air is drawn through the apparatus, displacing the hydrogen. Finally, disconnect the apparatus, and at once close the ends of the tube *D* (Fig. 26) with the rubber tubes provided with glass rods. Weigh the boat and contents; also the tube *D*. From your results calculate the composition of water.

In order that the experiment may be successful, it is absolutely essential that the current of hydrogen be maintained constantly until the apparatus is disconnected; otherwise the water formed will not pass over into the tube *D*.

**39. Hydrogen peroxide.** *a.* To about 50 cc. of water in a suitable flask add 3 or 4 g. of barium peroxide. Keep the flask cool with running water while slowly adding dilute sulfuric acid until the solution changes blue litmus paper to bright red. Filter off the precipitate (barium sulfate). The clear solution contains hydrogen peroxide.

*b.* The following is a test for hydrogen peroxide: Obtain about 1 cc. of starch paste (side shelf) and dilute to 5 cc. Add a very small crystal of potassium iodide (or 1 cc. of solution) and then add a few drops of the solution of hydrogen peroxide. An intensely blue color is produced.

*c.* Place about 2 g. of finely powdered manganese dioxide in a test tube and add 3 or 4 cc. of the solution of hydrogen peroxide. What evidence do you observe that a gas is evolved? Test it with a glowing splint. What is it?

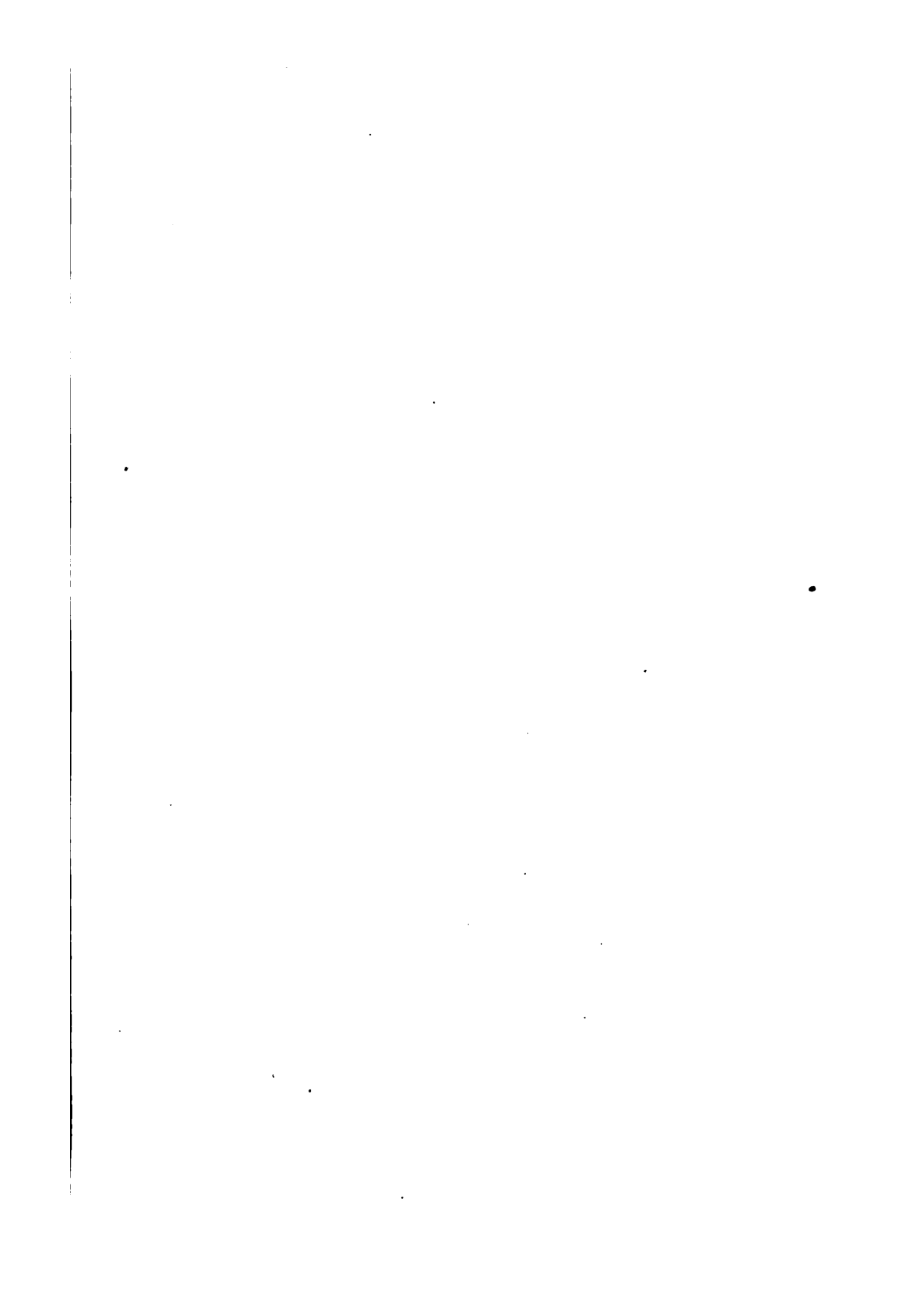


*d.* Repeat, using an equal bulk of powdered charcoal in place of the manganese dioxide.

*e.* To 1 or 2 cc. of a hot dilute solution of lead nitrate add a solution of hydrogen sulfide. A black precipitate of lead sulfide is produced. Boil a moment or two and then allow the precipitate to settle. Pour off the water, wash the precipitate once with hot water, and then add the solution of hydrogen peroxide. What change do you note in the color of the precipitate? How do you account for the change?

*f.* Pour 3 or 4 cc. of a solution of the coloring matter called cochineal in a test tube. Add 1 or 2 cc. of the solution of hydrogen peroxide and shake the contents of the tube vigorously. Is the color of the cochineal changed? What property of hydrogen peroxide is illustrated by this experiment?

*g.* Pour 3 or 4 cc. of the solution of hydrogen peroxide into a test tube and add rather more than an equal volume of ether. Shake the solution vigorously and notice that the ether quickly rises to a colorless layer on top of the solution. Now add 1 drop of a solution of potassium dichromate and again shake. Is the ether layer still colorless? A blue color in the ether is a delicate test for hydrogen peroxide.



## CHAPTER V

### THE STATES OF MATTER

**40. Evaporation.** Place about 1 cc. of ether or of chloroform on a watch crystal and blow upon the surface of the liquid. Why does it evaporate so fast? Do you notice any change in the temperature of the watch glass? Why is this?

**41. Boiling point.** Pour about 250 cc. of distilled water into a suitable flask or beaker, and heat it at such a rate that the temperature rises slowly but steadily. Use a thermometer to keep the water stirred. At what temperature do you observe bubbles? Where do they appear to form? What are they? At about what temperature do larger bubbles begin to form at the bottom of the vessel? What becomes of them? Why? At what temperature do they move freely up through the liquid to the surface? Do they get larger or smaller as they rise? Why? When the water is gently boiling, try increasing the heat. Is the boiling any more energetic? Does the temperature rise any? How do you define the boiling point?

**42. Freezing point; melting point. a.** Powder about 3 g. of sulfur and place it in a small test tube. Heat it very slowly a little above a small Bunsen flame, stirring it with a thermometer. At what temperature does the sulfur begin to melt? During the process of melting watch the thermometer closely. Does the temperature change during the process? When all the sulfur has melted, remove the flame and let the sulfur cool. At what temperature does it begin to solidify? Does the temperature change during solidification?

**b.** Repeat the whole experiment with a little paraffin. Do you notice any differences? To the melted paraffin add a little vaseline. How does this affect the melting point of the paraffin?





c. If the salts are available, determine the melting point of crystallized calcium nitrate, crystallized sodium acetate, crystallized sodium thiosulfate.

**43. Sublimation.** Place 2 or 3 g. of ammonium chloride in a small evaporating-dish and warm it gently. Does it melt? What change do you notice? Cover the dish with a small inverted funnel and continue to heat very gently so that not too much smoke issues from the stem of the funnel. After a time let the dish cool. Is there any solid upon the sides of the funnel? How did it get there? What is such a process called?



## CHAPTER VI

### THE LAWS OF CHEMICAL COMBINATION

#### I. THE LAW OF DEFINITE COMPOSITION

**44. Determination of the weight of oxygen which combines with a definite weight of magnesium.** *a.* Thoroughly clean and dry the porcelain crucible and lid included in your list of apparatus and weigh them accurately. Obtain a piece of magnesium ribbon weighing not to exceed 0.5 g. and scrape it with a knife until the surface is bright. Cut the ribbon into pieces 1 or 2 cm. in length, place the pieces in the crucible, and again weigh. Now place the covered crucible on the triangle and apply a gradually increasing heat. Magnesium burns violently when freely exposed to the air, hence the cover should be left on the crucible until the oxidation is nearly complete. This will require about twenty minutes. Cautiously remove the cover by means of the forceps, but hold it just above the crucible so that it can be returned at once in case the magnesium should begin to burn. Continue the heating until the substance no longer glows when the cover is removed. Finally, tip the crucible partly on its side, so as to give free access of air, and apply a strong heat for a few minutes longer. Withdraw the flame and reweigh the crucible, first allowing it to cool.

*b.* From your results calculate the weight of oxygen which combines with 1 g. of magnesium. Repeat the experiment, using a different weight of magnesium. In case the time is not sufficient to repeat the experiment, compare your result with those obtained by other members of the class. Are they in accord with the law of definite composition? If not, how do you account for their variation from the law?



**45. Determination of the percentage of oxygen in the black oxide of copper.** Use the apparatus shown in Fig. 26, except that a short glass tube is substituted for the tube *D*. Accurately weigh two porcelain boats, introduce different weights of black copper oxide, and reweigh. Place the boats in the glass tube and proceed with the experiment in accordance with § 38. Apply the heat first to the boat nearest the hydrogen generator. Continue the heating for fifteen or twenty minutes, in order completely to reduce the oxide. After the apparatus has cooled, weigh the boats and calculate the percentage of oxygen in each sample of the oxide. What do the results show?

**46. Determination of the weight of common salt obtained by adding hydrochloric acid to a definite weight of sodium bicarbonate.** When hydrochloric acid is added to sodium bicarbonate there are formed common salt (sodium chloride), water, and carbon dioxide. To determine the weight of salt formed from a definite weight of sodium bicarbonate, proceed as follows: Carefully weigh the evaporating-dish and watch glass. Transfer to the dish about 1 g. of sodium bicarbonate and reweigh. Pour 4 or 5 cc. of water on the bicarbonate, and place the watch glass on the dish so that only the lip of the dish remains uncovered. Now pour down the lip of the dish 2 or 3 drops of hydrochloric acid. Wait until the effervescence caused by the escape of the carbon dioxide ceases, then add a few drops more of the acid. Repeat until the addition of the acid no longer causes any effervescence. Now lift the watch glass and with a little water carefully rinse back into the dish the liquid which has collected on the undersurface of the watch glass. Lay the watch glass aside and slowly evaporate the solution, taking care that it does not quite boil (why?). When only a few drops of the liquid remain, cover the dish with the watch glass and increase the heat. The tip of the flame should just touch the bottom of the dish. Continue the heating until there is no more liquid left in the dish or clinging to the undersurface of the glass. Then withdraw the heat, and after the dish is cool, reweigh. The residue is



a rubber tube, which can be closed by the screw clamp *C*. Disconnect the bottles *A* and *B* at *D* and fill the bottle *B* and the exit tube with water, as in § 31. Weigh out accurately about 1 g. of zinc; then remove the cork from bottle *A* and transfer the zinc to the bottle. Also add to the bottle 1 drop of a solution of copper sulfate. Close the screw clamp *C*, and nearly fill the funnel with a dilute solution of sulfuric acid prepared as directed in § 23.

Place a beaker under the tube *F* and open the screw clamp until both the rubber and glass tubes are completely filled with the dilute acid; then quickly close the clamp. Connect the apparatus just as shown in the figure, care being taken to make the joints air-tight. Now adjust the pressure of the air inside the bottles,

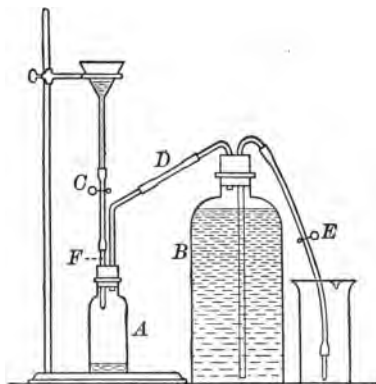


FIG. 27

after opening the clamp *E*, partially open the clamp *C*, and allow a few drops of the acid to flow into the bottle *A*. As the hydrogen is evolved it forces the water from bottle *B* into the beaker. More acid is added from time to time. After the zinc has all dissolved and the apparatus has acquired the room temperature, again adjust the pressure of the gas within the bottles and close the clamp *E*. Insert the values in the following table:

Weight of zinc taken . . . . .	
Volume of water forced into the beaker . . . . .	
Volume of liquid left in bottle <i>A</i> . . . . .	
Volume of hydrogen liberated . . . . .	
Temperature . . . . .	
Barometric reading . . . . .	

Reduce to standard conditions the volume of the hydrogen obtained and compare it with the theoretical results. What sources of error are involved in the experiment?





## CHAPTER VII

### CARBON AND CARBON DIOXIDE

**50. Carbon.** *a.* Arrange an apparatus as shown in Fig. 28. Partly fill the hard-glass test tube *A* with small pieces of wood—preferably with hardwood sawdust. Heat the wood, gently at first and then more strongly. After some liquid has collected in *B*, apply a light to the jet *C*. Are combustible gases evolved on distilling wood? Continue the heating as long as gas is evolved, then examine the liquid collecting in *B*. Is it soluble in water? How would you describe its odor? What is the residue left in *A*? Will it burn? Does it leave any ash?

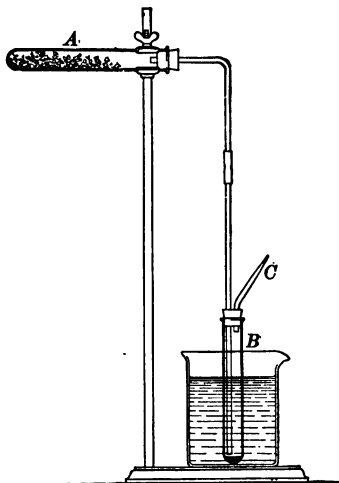


FIG. 28

*b.* Bring a cold porcelain dish into a small, luminous Bunsen flame. Note the deposit. What is this form of carbon called? In what other forms does carbon exist? What properties have all these forms in common?

*c.* Put one fourth of a test tube full of bone black into a small flask and pour over it about 50 cc. of water, to which has been added a few drops of a solution of litmus or of indigo. Thoroughly mix the contents of the flask, then heat it gently for a few minutes, and filter. If the filtrate is not



decolorized, repeat, using more bone black. What is the composition of bone black? By what other name is it known? What use does this experiment suggest for it?

*d.* Is carbon at ordinary temperatures an active element? Test it with the common acids. How does the charring of wood preserve it?

*e.* Prepare an apparatus according to Fig. 29. The bottle *A* contains a solution of sodium hydroxide, while *B* and *C* contain a solution of calcium hydroxide (limewater). The hard-glass tube *D* contains one or two small pieces of charcoal. Charge your oxygen generator as in § 15 and connect the rubber delivery tube with *E*. Now pass a *slow* current of oxygen through the apparatus, at the same time heating the charcoal in *D* until it just begins to glow. Describe the results. What do they prove? Why pass the oxygen through the solutions in *A* and *B*? What reactions take place in *D* and *C* (R)?

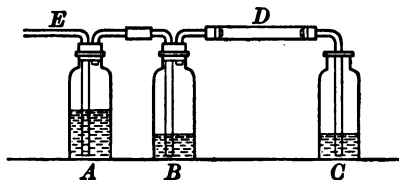


FIG. 29

*f.* In a hard-glass test tube heat an intimate mixture of 2 or 3 g. of black copper oxide and an equal bulk of powdered charcoal. Pass the evolved gases through a little limewater in a test tube. Write all the equations involved in the reactions. What remains in the test tube? How can you prove it? What use does this suggest for charcoal?

**51. Carbon dioxide.** *a.* Put some pieces of marble in your hydrogen generator, cover them with water, and add a little concentrated hydrochloric acid through the funnel tube. Fill two or three bottles with the gas evolved by downward displacement. (To test whether the bottles are filled or not, hold a burning splint in the mouth of the bottle.)

*b.* Devise an experiment to show whether the gas is heavier or lighter than air. Attempt to pour it from one bottle to another, as you would a liquid, and test with a burning splint for its presence in the second bottle.



c. Half fill a small beaker with limewater and pass carbon dioxide through the liquid (R). The formation of a white precipitate in limewater is a test for carbon dioxide.

d. By blowing through limewater prove the presence of carbon dioxide in the air exhaled from the lungs (R). Hold a wide-mouthed bottle above a small flame so that the hot gaseous products of combustion will collect in it; then quickly add a few cubic centimeters of limewater, cover the mouth of the bottle with the hand, and shake up the contents. What do the results prove? What are the sources of carbon dioxide in the air?

**52. Weight of 1 liter of carbon dioxide.** Obtain a 250-cc. Erlenmeyer flask (Fig. 30) and a well-rolled cork to fit it tightly. Insert a small glass tube through the cork. A piece of pure rubber tubing is slipped over the end of the glass tubing and the rubber tubing is closed with a pinch clamp as shown in the figure. Thoroughly dry the flask both inside and out, then insert the stopper tightly and mark the point to which it extends in the flask by a strip of paper cut from a label. Open the clamp for a moment to adjust the pressure of the air. Accurately weigh to third decimal place the flask and stopper, recording the reading of the barometer and the temperature of the air. Next arrange an apparatus as shown in Fig. 31. *A* represents the carbon dioxide generator, *B* represents a bottle partially filled with sulfuric acid, and *C* represents the 250-cc. flask (cork removed). Now generate a slow current of carbon dioxide in *A*. The gas is dried as it bubbles through the sulfuric acid in *B* and is then conducted into *C*, gradually displacing the air in *C*. Continue passing the gas into *C*, until you are satisfied that all the air has been expelled; then while the gas is still being evolved, slowly remove the tube *D* from the flask, and at once insert the cork in the flask to the point indicated by the gummed paper. Open the clip for a moment to adjust the pressure; then weigh the flask as

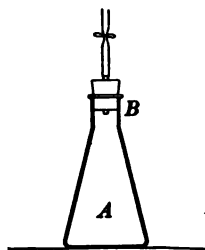


FIG. 30



before. Next completely fill the flask with water and insert the cork, allowing the extra water to run out through the rubber tubing; then measure the water by transferring it to

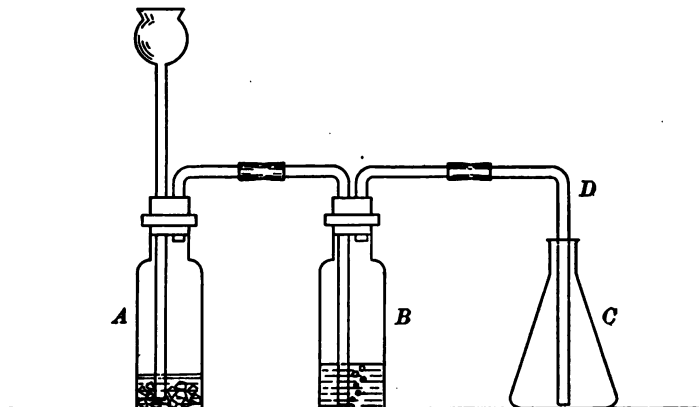


FIG. 31

a graduated cylinder. This will give you the volume of the flask. Insert the appropriate values in the table below and calculate the weight of 1 liter of carbon dioxide.

1. Barometric pressure . . . . .
2. Temperature . . . . .
3. Volume of flask . . . . .
4. Weight of flask filled with air under laboratory conditions . . . . .
5. Weight of 1 liter of air under standard conditions . . . . . 1.293 g.
6. Weight of 1 liter of air under laboratory conditions . . . . .
7. Weight of flask empty. (Calculate from 3, 5, and 6) . . . . .
8. Weight of flask filled with carbon dioxide under laboratory conditions . . . . .
9. Weight of carbon dioxide in flask . . . . .
10. Weight of 1 liter of carbon dioxide under standard conditions. (Calculate from 3 and 9) . . . . .





## CHAPTER VIII

### NITROGEN AND THE ATMOSPHERE

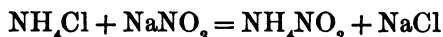
**53.** The preparation and properties of nitrogen. (*On account of its great affinity for oxygen, phosphorus must be kept and handled under water. Never bring the dry substance in contact with the skin, as it may ignite and cause a serious burn.*)

**a.** Cover the bottom of a pneumatic trough with water to a depth of 2 or 3 cm. Float on the water a porcelain crucible containing a small piece of phosphorus. Ignite the phosphorus by touching it with the hot end of a wire or file, and quickly invert over the crucible a large beaker or wide-mouthed bottle, being careful to keep the rim of the beaker below the surface of the water. The white fumes formed have the composition  $P_2O_5$ . Leave the beaker in position until the fumes have entirely disappeared. Note that the water has risen in the beaker. Explain. Adjust the beaker or the water in the trough so that the level of the liquid inside and outside of the beaker is the same; then cover the beaker with a glass plate and turn it into an upright position. Test the gas with a burning splint.

**b.** In a 250-cc. flask place a mixture of 3 g. of ammonium chloride and 6 g. of sodium nitrite, and add 20 cc. of water. Provide the flask with a cork (one-hole) and delivery tube, so that the gas evolved may be collected over water as in the case of oxygen and hydrogen. Have at hand a vessel of cold water so that the flask may be cooled by lowering it into the water in case the action becomes too violent. Clamp the flask on a ring stand and apply a *very gentle* heat, moving the burner about with the hand. As soon as the action begins, withdraw the burner. After the air has been expelled from



the apparatus fill two or three bottles (250-cc.) with the gas. If the action becomes too violent, immerse the flask in cold water. The reaction which takes place is expressed in the following equation:



The ammonium nitrite then decomposes into water and nitrogen (R). (The symbol (R) indicates that the equation for the reaction is to be written.) Note the physical properties of the gas. Test with a burning splint.

**54. Determination of the relative volumes of nitrogen and of oxygen in the air.**<sup>1</sup> This determination may be made by bringing in contact with a definite volume of air a liquid which not only absorbs the oxygen but in doing so flows into the tube which contains the air and fills a space equal to that previously occupied by the oxygen. The volume of this liquid can be easily measured, and in this way the volume of the absorbed oxygen may be ascertained.

The solution used to absorb the oxygen soon loses its strength on exposure to the air; hence the experiment must be performed rapidly. Before preparing the solution the student should practice the manipulations involved in the experiment.

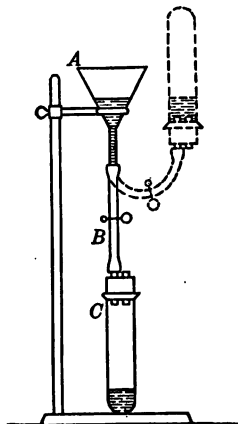


FIG. 32

Prepare an apparatus according to Fig. 32. In this apparatus *C* represents a test tube about 15 cm. in length (use the hard-glass tube employed in the preparation of oxygen). The tube is fitted with a two-hole rubber stopper. Through one of the holes is fitted a glass tube, which, together with the accompanying rubber tube and funnel, is the same as was used to introduce the acid into the bottle *A* (Fig. 27). The remaining hole in the stopper is closed with a glass rod. Notice that

<sup>1</sup> From Cooley's "Laboratory Studies."



both the glass rod and the tube extend just through the small end of the stopper. Now close the rubber tube tightly with a screw clamp *B*. Disconnect the test tube and remove the glass rod from the stopper, preparatory to performing the experiment. Prepare an alkaline solution of pyrogallic acid, as follows: Dissolve 5 g. of potassium hydroxide in 5 cc. of water and cool the solution to room temperature. Add this to a solution of 4 g. of pyrogallic acid in 10 cc. of water, and *at once* pour the resulting liquid into the funnel *A*. Quickly open the screw clamp until both the rubber tube and the glass tube are filled with the liquid, then close tightly. Connect the test tube, holding it by the rim to avoid heating the contained air, and insert the glass rod in the cork. The air inclosed in the tube is now at the same temperature and pressure as the surrounding air. Now open the screw clamp. The liquid flows in, absorbing the oxygen. When the liquid ceases to enter, grasp the tube by the rim and invert it as shown in the dotted lines of the figure, adjusting it so that the level of the liquid is the same in both the tube and the funnel (why?). Then clamp the rubber tube and return the test tube to its original position. Mark the volume of the air originally inclosed in the tube by placing a narrow strip of gummed paper about the tube at the lower end of the stopper; also mark by a strip of paper placed at the level of the liquid in the tube, the volume of the oxygen absorbed. Disconnect the tube and rinse it. Measure the volume of the tube to each strip of paper by pouring in water from a graduated cylinder. From these data calculate the volume of oxygen and of nitrogen in 100 volumes of air.

NOTE. This experiment disregards the presence in air of all constituents other than oxygen and nitrogen. The volume of such constituents, however, in the volume of air taken, is so small that it may be neglected.

- 55. Other constituents of the air.** *a.* Expose a piece of calcium chloride to the air for two or more hours. Explain.
- b.* Expose a few cubic centimeters of limewater to the air for a half hour. Explain the results.



## CHAPTER IX

### SOLUTIONS

**56. Rate of solution.** *a.* Drop a crystal of potassium permanganate into a test tube partly filled with water and shake the liquid until the solid is dissolved. Note the color of the solution.

*b.* Place a test tube filled with water in a test-tube rack and drop into it a crystal of potassium permanganate. Allow the liquid to remain (without shaking it) until near the close of the laboratory period. Note the result. What does the experiment show?

**57. Solubility.** *a.* To a test tube two thirds full of boiling water add common salt, small portions at a time, as long as any dissolves. If an excess remains undissolved after boiling a short time, add just enough water to bring it into solution. Immerse the test tube in a beaker of cold water. Is salt more soluble in cold or in hot water? Repeat the experiment, using powdered potassium chloride or potassium nitrate instead of salt, immersing the test tube in the same beaker as before. When the two tubes have cooled, note the relative quantities of the solids that have separated. What inference do you draw?

*b.* Filter the salt solution through a dry filter into a dry test tube and note the temperature. Weigh a small evaporating-dish and watch-glass cover; then pour 10 or 15 cc. of the clear salt solution into the dish and reweigh. Now evaporate to dryness, taking note of the precautions given in § 46. When the dish is cool, reweigh. From your results calculate the solubility of common salt at the indicated temperature.

*c.* In a similar way determine the solubility of the potassium nitrate or of potassium dichromate.





**58. Supersaturated solutions.** To 20 cc. of water in a beaker add 20 g. of hydrated sodium sulfate and gently warm until solution is complete. Consult the solubility curve of sodium sulfate. How high is it advisable to heat the solution? Is the solution mobile or viscous? Pour the solution into two test tubes and set it aside to cool. If no crystallization occurs at room temperature, the solution should be supersaturated. Add a very small crystal of the solid sodium sulfate. Why do crystals form? In what condition is the remaining solution? Define supersaturation. A concentrated solution of ferric nitrate in dilute nitric acid saturated at about 60° is more certain than the hydrated sodium sulfate to work well.

**59. Solubility of liquids.** Pour about 5 cc. of water into a test tube, add an equal volume of kerosene, and mix the liquids by shaking the test tube. Set the tube aside for a few minutes and note the result. Repeat the experiment, substituting alcohol for kerosene. Note the result.

**60. Fractional distillation.** Distill a mixture of 10 cc. of alcohol (boiling point 78.3°) and 30 cc. of water. Collect the first 1 or 2 cc. of the distillate in an evaporating-dish and test with a flame. In the same way test successive portions of the distillate. Does there seem to be a separation of the two liquids? By the above method a mixture of liquids boiling at different temperatures may often be separated more or less perfectly. What name is given to this process?

**61. Constant-boiling solutions.** To 1 or 2 cc. of dilute hydrochloric acid add a drop or two of a solution of silver nitrate. In this case the formation of a white precipitate (silver chloride) shows the presence of hydrochloric acid in the solution. Pour about 75 cc. of dilute hydrochloric acid into a distilling-flask connected with a Liebig condenser and slowly distill, catching the distillate in a test tube. When about 5 cc. has been collected, test it with 1 or 2 drops of silver nitrate solution. Is there an appreciable precipitate? Continue the distillation, testing each portion of 5 cc. as it is collected. Does the quantity of the precipitate formed increase gradually or suddenly? When a decided precipitate is obtained, collect about 2 cc. of



the distillate and set it aside; then, without further testing, distill over as much of the solution as you safely can, collecting the last 2 cc. See that these two portions are as nearly equal as possible, then dilute each to about 15 cc., and to 5 cc. of each add silver nitrate as long as a precipitate forms. Allow the precipitate to settle, and compare the two quantities. From all of your observations do you think it would be possible to fraction the more concentrated solutions of hydrochloric acid?



## CHAPTER X

### CHLORINE; HYDROGEN CHLORIDE; HYDROCHLORIC ACID

**63. Preparation of chlorine.** (*All the following experiments must be performed in the hood, and great care taken not to inhale the gas.*) *a.* Place about 1 g. of manganese dioxide in a test tube, add 2 or 3 cc. of hydrochloric acid, and heat *gently* (R).

*b.* In a similar way test the action of hydrochloric acid upon lead peroxide, potassium permanganate, and *potassium* dichromate. Will all compounds containing oxygen oxidize hydrochloric acid? Try lead oxide (litharge) and sodium sulfate.

*c.* Repeat experiment *a*, replacing the hydrochloric acid with small amounts of common salt and sulfuric acid. The manganese dioxide and salt should first be mixed together and the sulfuric acid added to the mixture. Compare the reactions in *a* and *c* (R). Which of the two methods is the cheaper for preparing chlorine? Give reason for your answer.

*d. Usual laboratory method* (two students working together). Arrange an apparatus according to Fig. 33, using a 250-cc. flask. Thoroughly clean and dry five wide-mouthed bottles (250-cc.) for collecting the gas. Place in the flask from 20 to 25 g. of manganese dioxide. Insert the cork and pour 150 cc. of hydrochloric acid through the funnel tube. Shake the flask so as to thoroughly wet the

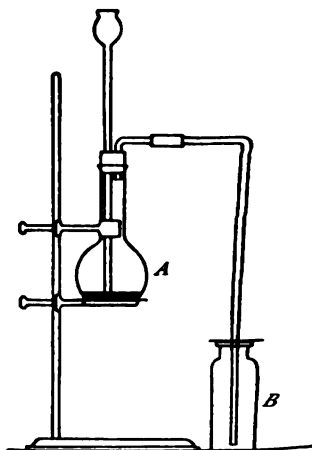


FIG. 33

YDROCHLOR

1/1/1  
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1/1/1

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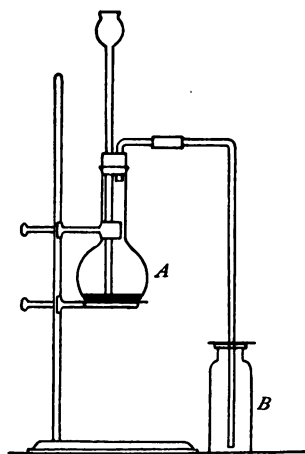


FIG. 33





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*d. Usual laboratory method* (two students working together). Arrange an apparatus according to Fig. 33, using a 250-cc. flask. Thoroughly clean and dry five wide-mouthed bottles (250-cc.) for collecting the gas. Place in the flask from 20 to 25 g. of manganese dioxide. Insert the cork and pour 150 cc. of hydrochloric acid through the funnel tube. Shake the flask so as to thoroughly wet the

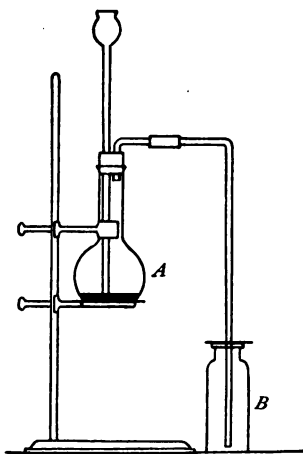
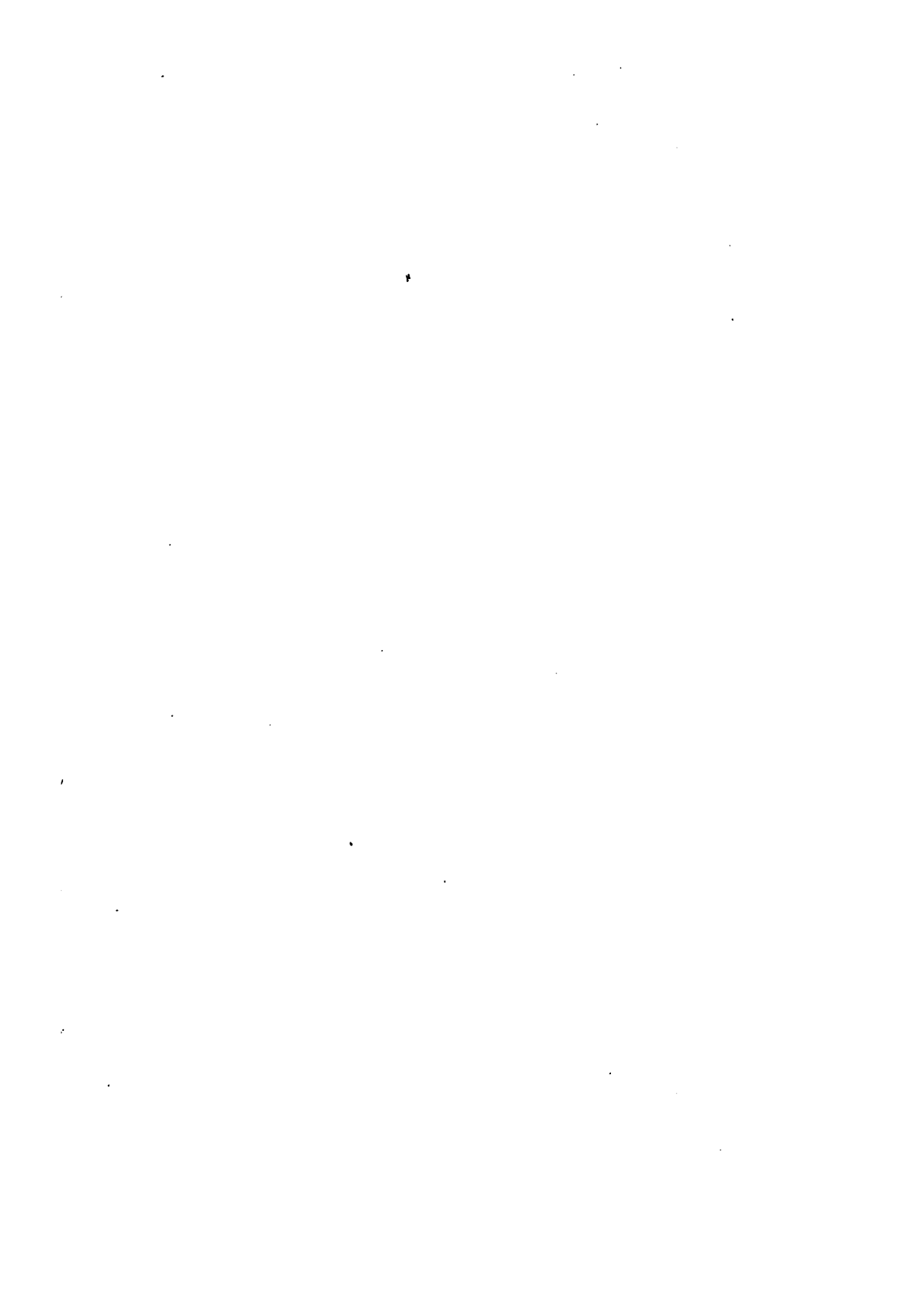


FIG. 33



## CHAPTER X

### CHLORINE; HYDROGEN CHLORIDE; HYDROCHLORIC ACID

**62. Preparation of chlorine.** (*All the following experiments must be performed in the hood, and great care taken not to inhale the gas.*) *a.* Place about 1 g. of manganese dioxide in a test tube, add 2 or 3 cc. of hydrochloric acid, and heat gently (R).

*b.* In a similar way test the action of hydrochloric acid upon lead peroxide, potassium permanganate, and potassium dichromate. Will all compounds containing oxygen oxidize hydrochloric acid? Try lead oxide (litharge) and sodium sulfate.

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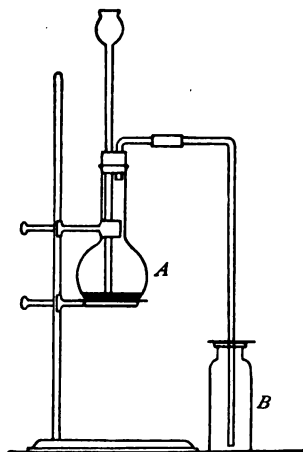
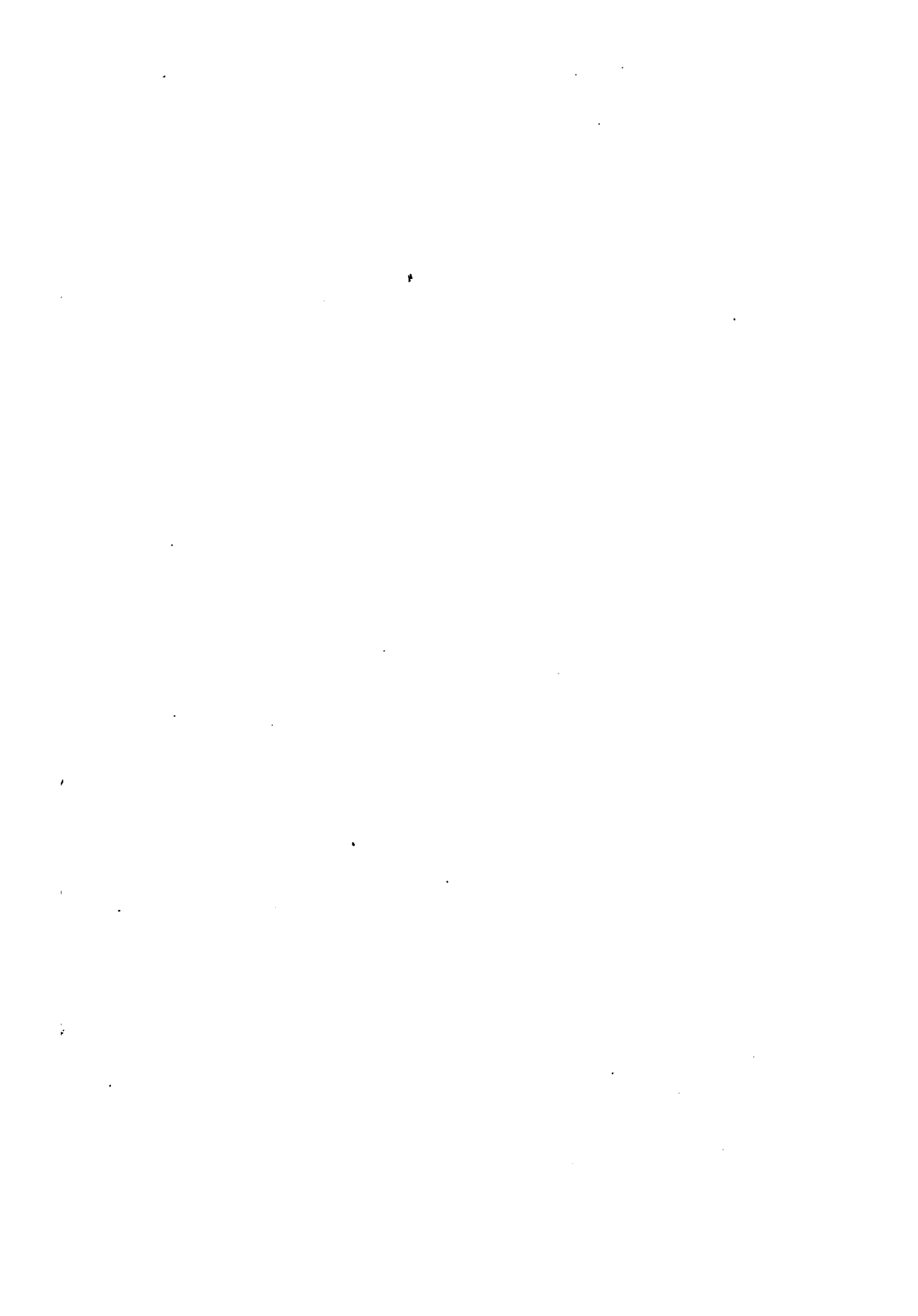


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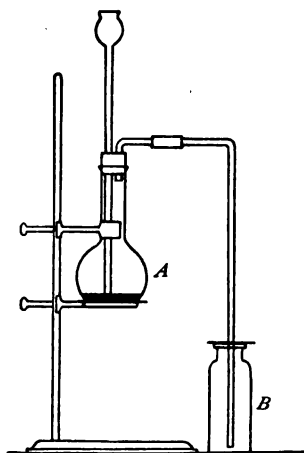


FIG. 33



manganese dioxide. Warm gently, applying just enough heat to cause a gentle evolution of the gas but not enough to boil the liquid. Fill all the bottles with the gas, collecting it by downward displacement; then prepare some chlorine water by bringing the exit tube into a bottle containing a little water, so that the gas bubbles up through the liquid. Continue the heating until no more chlorine is evolved (chlorine water).

*e.* A part of the class should prepare chlorine as follows: Instead of the funnel tube (Fig. 33) use a dropping-funnel (an ordinary funnel connected with a straight glass tube by a piece of rubber tubing may be used, a screw clamp on the rubber connection taking the place of the stopcock, as shown in Fig. 17). Place about 20 g. of potassium permanganate crystals in the dry flask and slowly admit concentrated hydrochloric acid from the dropping-funnel, regulating the flow of the acid by the rate at which chlorine is evolved.

**63. Properties of chlorine.** *a.* Sprinkle a pinch of finely powdered antimony into one of the bottles of the gas.  $\text{SbCl}_3$  is formed. Is this reaction an example of combustion? Is the presence of oxygen necessary for combustion?

*b.* Support by forceps a small piece of copper foil, heat it to redness, and *immediately* drop it into a bottle of the gas. Describe the result. What is formed?

*c.* In a bottle of the dry gas suspend a strip of colored calico and two strips of paper, one with dry writing in ink on it, the other with printing (printer's ink).

*d.* Repeat *c*, using similar strips moistened with water. Describe the results in *c* and in *d*. What part does the water play in the bleaching?

*e.* Test the action of the gas on strips of red and of blue litmus paper. Generate hydrogen by the usual method. After taking the required precautions (what are they?) ignite the hydrogen delivered from a jet and introduce the flame into a bottle of chlorine. Does the hydrogen continue to burn? What is formed? Allow the hydrogen to burn until the chlorine is all used up; then test with moist litmus paper(?).





#### 64. Preparation of hydrogen chloride and hydrochloric acid.

Connect the generator used in the preparation of chlorine (Fig. 33) with a wide-mouthed bottle, as shown in Fig. 34. The delivery tube *F* is cut at *A* and at *D*, and the ends (rounded) are joined by rubber tubing. Pour about 30 cc. of water into the bottle *B*. Notice that the tube extending into

the bottle *does not quite* touch the surface of the water. Prepare some dilute sulfuric acid by carefully pouring 25 cc. of the concentrated acid into 10 cc. of water (note precaution, § 23). The mixture must be stirred and cooled from time to time while the acid is being added. Have at hand two *dry* bottles in which to collect some of the gaseous hydrogen chloride. Put about 50 g. of common salt into the generator flask, insert the cork, pour the cold dilute sul-

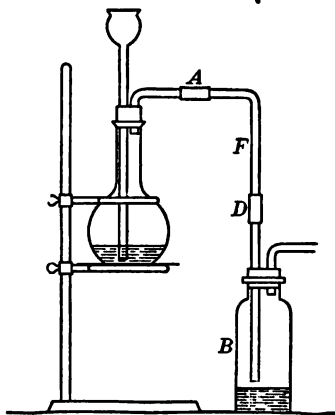


FIG. 34

furic acid through the funnel tube, mix the contents by a gentle motion of the flask, and after two or three minutes warm gently with a *small* flame. Notice the currents in the water in *B*. What is the cause of them? As soon as the gas is evolved regularly, disconnect the generator flask at *D* long enough to collect (by downward displacement) two bottles of the gas. Cover these tightly with glass plates and set aside; then again connect the generator with *B* and continue to apply a gentle heat as long as any gas is evolved. What two reactions are possible? From the weights of materials used and the conditions of the experiment, which one would you expect to take place? Why not extend the tube in *B* to the bottom of the bottle?

**65. Properties of hydrogen chloride.** *a.* What is the color of gaseous hydrogen chloride. Account for the formation of fumes when the gas is exposed to the air.



*b.* Test with a lighted splint one of the bottles of gas collected in the experiment of § 64. Is it combustible? Is it a supporter of combustion?

*c.* Fill a large beaker with water and color it with a few drops of a solution of blue litmus. Now uncover the remaining bottle of gas collected in the experiment of § 64, invert it, and at once bring the mouth of it under the surface of the water in the beaker. Describe the results. What does the experiment prove?

**66. Properties of hydrochloric acid.** *a.* Put a drop of the aqueous solution of the acid from bottle *B* (§ 64) on a bit of blue litmus paper. Note the result. Add two drops of the solution to 3 or 4 cc. of water and taste a drop. Make a test-tube experiment to prove the presence of chlorine in the acid; also one to prove the presence of hydrogen. Write the equations in each case. How does the solution compare with the hydrochloric acid on your desk?

*b.* Pour out some of the solution from the generator flask into a beaker and allow it to cool. Add some concentrated hydrochloric acid. What change do you note? Is this reaction reversible?

*c.* For the determination of the composition of hydrogen chloride, see § 113.



## CHAPTER XI

### SODIUM ; SODIUM HYDROXIDE

**67. Sodium ; sodium hydroxide.** *a.* Recall § 20. Obtain a small piece of sodium from the instructor. Cut it and note the rapidity with which the freshly cut surface is tarnished. Half fill an evaporating-dish with water, then drop the sodium into it and quickly cover the dish with a glass plate. After the action (R) has entirely ceased, test the liquid with red litmus paper. What compound is dissolved in the water?

*b.* Weigh out about 5 g. of good quicklime and moisten it with about an equal weight of water in an evaporating-dish, warming it, if necessary, to make it slake (R). When it has ceased slaking, dilute to 100 cc. and add the required weight of sodium carbonate to produce complete double decomposition (R). Boil for a few minutes to render filtration easier, and then filter. What is the filtrate? How is this obtained commercially in solid form? Test a little of the filtrate to see if an excess of carbonate has been added (how will you do this?), and if an excess is found, devise a way to remove it. Add 2 drops of the filtrate to 5 cc. of water and taste the solution (?). Dip a piece of red litmus paper into the filtrate (?).



## CHAPTER XII

### ACIDS; BASES; SALTS; NEUTRALIZATION

**68. Acids.** *a.* Recall the properties of hydrochloric acid (§ 66). Prepare dilute solutions of each of the following acids by adding 2 cc. of each of the concentrated acids to 10 cc. of water and thoroughly mixing: hydrochloric, nitric, sulfuric, acetic. By means of a clean glass rod transfer a drop of each to a piece of blue litmus paper and then to a piece of red. What changes do you notice?

*b.* Recall the action of hydrochloric, sulfuric, and nitric acids on zinc (§§ 22, 23). Nitric acid is a strong oxidizing agent. Might this account for the fact that hydrogen is not evolved when this acid reacts with zinc?

*c.* Add one drop of hydrochloric acid solution to 10 cc. of water. Stir thoroughly and taste the solution. Repeat, using acetic acid (?). Compare the formulas of the acids. In what respect are the acids similar in composition?

**69. Bases.** Prepare dilute solutions of each of the following bases: sodium hydroxide, potassium hydroxide, calcium hydroxide. Try the effect of each of these solutions on blue and on red litmus paper. Taste a drop of the calcium hydroxide solution. Compare the formulas of the above bases. In what respects are the bases similar in composition? From your results characterize the properties of bases on the supposition that the above compounds are typical bases.

**70. Salts.** Dilute 5 cc. of the ordinary laboratory solution of sodium hydroxide (1 part of the solid to 10 parts of water) with an equal volume of water. To this solution add a few drops of hydrochloric acid; the two react with evolution of heat (R). Stir the resulting solution with a glass rod and test its action on blue and on red litmus paper. Has it acid





or basic properties? Continue to add the acid, drop by drop, with stirring, till the resulting solution is neutral or, at most, slightly acid. Pour the solution into an evaporating-dish and evaporate to dryness. What compound remains? What is the name given to the compounds formed by the action of acids with bases? Characterize these compounds.

**71. Ratio of acid to base in neutralization.** Prepare a dilute solution of sodium hydroxide by diluting 20 cc. of the laboratory reagent to 100 cc.; also a dilute solution of sulfuric acid by adding 1 cc. of the concentrated acid to 100 cc. of water. Rinse out a burette (storeroom), first with distilled water, then with a little of the alkaline solution. Support the burette (Fig. 35) and pour the alkaline solution into it until the level of the liquid is 2 or 3 cm. above the zero mark. Turn the stopcock and let the solution flow out until the bottom of the curved surface (meniscus) is on a level with the zero mark. In a similar way fill a second burette with the acid solution. Now let exactly 15 cc. of the acid solution flow into a small beaker, add 2 drops of a solution of phenolphthalein and run in 2 or 3 cc. of the alkaline solution. Notice that where the liquids come in contact, a reddish color is produced, which disappears quickly on stirring. Run in more of the solution, a little at a time, until the color fades slowly, then a drop at a time until the entire liquid, on stirring, remains colored *faintly* red. This marks approximately the point of neutralization. Note the number of cubic centimeters of the alkaline solution used. Repeat the experiment, using different volumes of acid, say 10 cc. and 20 cc. Calculate in each case the number of cc. of the alkaline solution required to neutralize 1 cc. of the acid solution. What do the results prove?

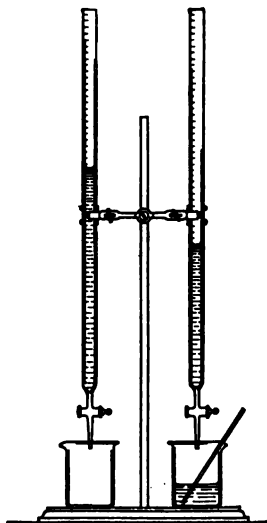


FIG. 35



## CHAPTER XIII

### IONIZATION

**72. Conducting power of various solutions.** *a.* Obtain a conductivity apparatus (Fig. 36) from the storeroom and polish the copper-wire electrodes with emery paper until they are bright and free from oxide. At the beginning of each experiment see that the electrodes are bright and dry, and

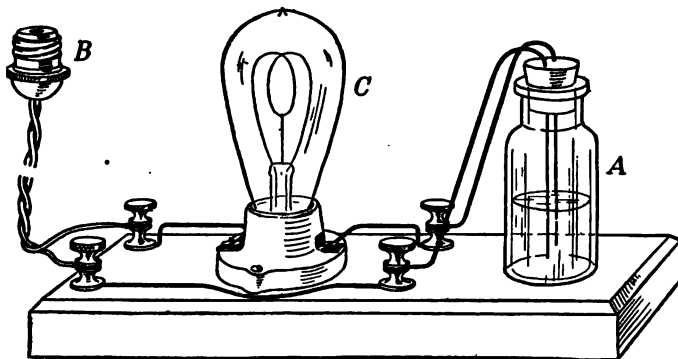


FIG. 36

that the cell *A* is also perfectly clean and dry. Unscrew a lamp *C* from a convenient socket above your desk, screw it *loosely* into the socket on your apparatus, and attach the apparatus to the empty socket on the lighting system by means of the extension cord and plug *B*. Every time a change is to be made in the cell, loosen the lamp in the socket, and do not screw it down to make contact until all the connections of the cell have been arranged.

*b.* Partly fill the cell *A* with dry powdered salt, dip the electrodes into the powder, arrange the connections at the



binding-posts, and screw down the lamp *C*. Have you any evidence that the powdered salt is a conductor?

*c*. In a similar way test the conductivity of distilled water and tap water.

*d*. Dissolve about 1 g. of the salt in 10 cc. of distilled water and test the conductivity of the solution. Account for the result as compared with the results obtained in *b* and *c*.

*e*. Repeat *b* and *d*, substituting sugar for salt. Account for any difference in results.

**73. Acids from the standpoint of the ionization theory.**

*a*. Test the conductivity of dilute solutions of the following acids: hydrochloric, sulfuric, acetic.

*b*. Ask the instructor for a solution of dry hydrogen chloride in benzene or toluene. Determine whether it conducts the electric current (care must be taken to exclude all moisture). Pour a little of the solution on a clean, dry iron nail. Repeat, using the ordinary aqueous solution. Account for the difference in action between the two solutions. Characterize acids from the standpoint of the ionization theory on the basis that the above acids are typical ones and that all others act like them.

**74. Relative strength of acids.** *a*. Obtain from your instructor about 15 cc. of twice normal solutions of each of the following acids: hydrochloric, sulfuric, and acetic. Test their relative conductivities (Fig. 36) by noting the brilliancy of the light evolved in each case (three students should work together, each using a different acid). Note the results.

*b*. From the standpoint of the ionization theory, upon what does the relative strength of two acids in equimolecular concentration depend? How will this affect the rate at which the two acids will act upon a third substance? Will the two acids, when treated with a metal, evolve hydrogen at the same rate? Will the volume of hydrogen ultimately evolved be the same? Test your statements by determining the rate at which each of the three acids used in *a* will give up hydrogen when brought in contact with zinc. To do this proceed as follows:



Arrange a hydrogen generator *A* (Fig. 37) and fit it with a delivery tube *B*. Fill a test tube *C* with water and invert it in a beaker of water, placing a slender rubber band around it about 5 cm. from the closed end. Weigh accurately 2 g. of granulated zinc and place it in *A*. Through the funnel tube add 10 cc. of twice normal hydrochloric acid, to which 2 drops of cupric sulfate has been added (why?), and bring the end of the delivery tube *B* under *C* so as to collect the evolved hydrogen. At once note the exact time to the second. Keep the rubber band at the level of the water in the beaker, and when the hydrogen has displaced the water in *C* to this level, again read the time to the second. Repeat the entire experiment, substituting twice normal sulfuric acid for the hydrochloric acid. Again repeat, using twice normal acetic acid. Compare the time required in the three experiments. What do you conclude as to the relative strengths of the three acids?

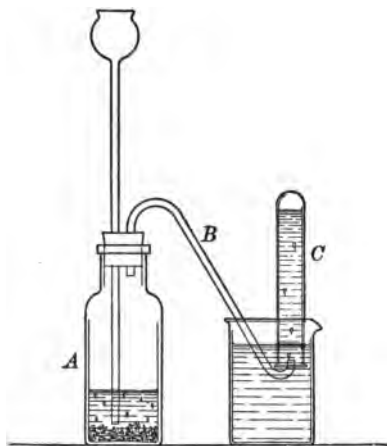


FIG. 37

*c.* Repeat the three experiments in *b*, using pieces of magnesite ( $\text{MgCO}_3$ ) of equal size instead of the zinc. Gaseous carbon dioxide ( $\text{CO}_2$ ) is evolved in this reaction. What is now the order of the three acids in strength?

**75. Bases and salts from the standpoint of the ionization theory.** *a. Bases.* Test the conductivity of dilute solutions of the following typical bases: sodium hydroxide, calcium hydroxide, potassium hydroxide. Discuss the results.

*b. Salts.* Test the conductivity of dilute solutions of the following typical salts: sodium sulfate, ammonium chloride, sodium acetate. Discuss the results.





**76. Color of ions and of molecules.** Prepare a small amount of dilute solutions of each of the following: potassium bromide, potassium sulfate, potassium chloride. From the standpoint of the theory of ionization, what ions are present in each of the solutions? What is the color of each of these ions? Place in a test tube about one-half gram of each of the following substances: cupric bromide, cupric sulfate, cupric chloride. Note the color of each. Now dissolve each in the smallest possible amount of water. Note the color of the solutions. Now gradually dilute each until the tubes are nearly filled with water. Mix each thoroughly and note the color of the solutions. Give an explanation of the results.

**77. Electrochemical series.** *a.* Dissolve about 1 g. of copper sulfate in a test tube two thirds full of water and dip an iron nail into the solution, first rubbing the nail bright with sandpaper (R). Reverse the experiment by dipping a bright piece of copper foil into a solution of ferrous sulfate. Is the reaction reversible? Why?

*b.* Suspend a piece of mossy zinc or tin by a thread in a solution of lead acetate, and let it stay undisturbed, noting the appearance of the metal from time to time. Would you expect a piece of silver or bismuth to give similar results?

*c.* Polish a piece of aluminium foil and dip it for a time in pure water. Is hydrogen evolved? Would you expect it to be? Now dip the foil into a dilute solution of mercuric chloride and watch for evidences of evolution of hydrogen. Is mercury deposited on the foil? Would it evolve hydrogen? A deposit of mercury prevents any oxide or hydroxide from sticking to the aluminium. Does this help in explaining the evolution of hydrogen?



## CHAPTER XIV

### COMPOUNDS OF NITROGEN

**78. Preparation of ammonia.** *a.* Dissolve 1 g. of ammonium chloride in a little water in a test tube and heat to boiling. Can you detect the odor of ammonia? Add a few drops of a solution of sodium hydroxide. Is the ammonia liberated (R)? Moisten a small strip of red litmus paper and hold it at the mouth of the tube. Note the result. Hold the end of a glass rod moistened with concentrated hydrochloric acid in the mouth of the tube. What is formed (R)?

*b. Usual laboratory method.* This differs from the method used in *a* only in the fact that the less expensive calcium hydroxide (slaked lime) is substituted for sodium hydroxide. Fig. 38 shows the form of apparatus used. *A* represents a 250-cc. flask; *B* and *C* are 250-cc. wide-mouthed bottles partly filled with water. Notice that the glass tubes extending into the bottles do not quite touch the surface of the water. Why? Two *dry* bottles will also be needed for collecting some of the gas.

Put into the flask *A* an intimate mixture of about 30 g. of finely powdered slaked lime and 15 g. of ammonium chloride; place it on a sand bath and heat gently with a *small* flame. A wire gauze may be

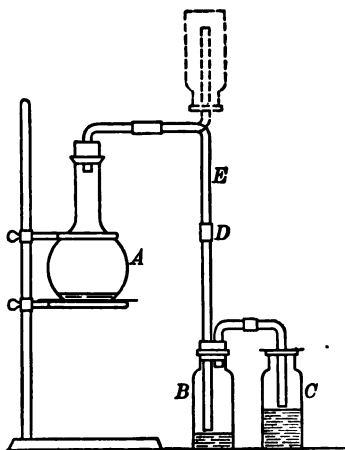


FIG. 38



substituted for the sand bath, provided the flask is clamped so that the bottom of it does not quite touch the gauze. In this case, however, the burner should be held in the hand and moved about so as to apply the heat uniformly, as otherwise the flask may be broken. As soon as the gas is evolved freely, disconnect the tube *E* at *D*, bring it to an upright position, as shown in the dotted lines, and collect two bottles of the gas by bringing them successively over the exit tube. To tell when they are filled, test for the presence of ammonia at the mouths of the bottles with a piece of red litmus paper moistened with water. As soon as the bottles are filled, cover them tightly and set them aside, mouth downward; then quickly connect the tube *E* with the bottle *B* again and continue to heat the mixture gently as long as any gas is generated. What is the source of each of the materials used in the preparation of the gas? Write the equations for all of the reactions involved.

**79. Properties of ammonia.** *a.* Note the color and odor of the gas. Is it heavier or lighter than air?

*b.* Test a bottle of the gas with a burning splint. Describe the results.

*c.* Fill a large beaker with water and color it with a few drops of red litmus solution. Uncover the remaining bottle of the gas and at once bring the mouth of it under the surface of the water in the beaker. What do the results prove?

*d.* Note the odor of the liquid in the bottle *B*. Try its effect on blue and on red litmus paper. How does it compare with the so-called "aqua ammonia" of the druggist? Does the gas combine with the water, or is it simply dissolved in it? Give reasons for your answer. Now neutralize the liquid with hydrochloric acid (*R*) and evaporate just to dryness. What is the residue? Test it with a few drops of sodium hydroxide solution. How does it compare with one of the compounds used in the preparation of the ammonia? Distinguish between the terms *ammonia* and *ammonium*.

*e.* Advanced students may at this time determine the composition of ammonia according to § 114.



**80. Nitrides.** Connect the delivery tube *E* (Fig. 38) with the hard-glass tube *C* (Fig. 26). In the middle of the tube place a shallow layer of magnesium powder about 2 cm. in length. Conduct ammonia through the tube, and when the air has been displaced, heat the tube under the magnesium strongly until the magnesium is red hot. Then increase the current of ammonia. What change takes place in the

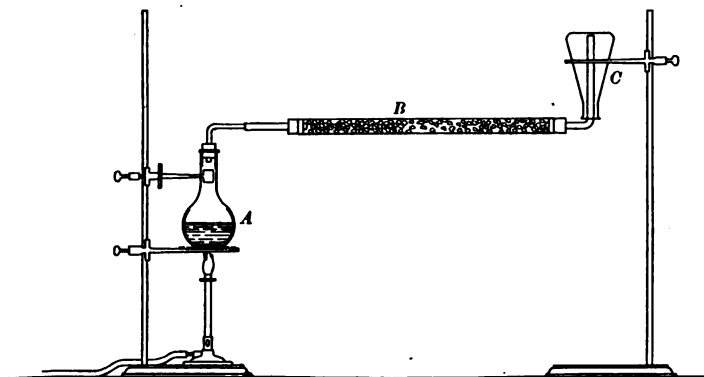


FIG. 39

magnesium (R)? When it is cool examine the powder. Place a little of it on a watch glass and moisten it with water. Can you detect the evolution of ammonia (R)?

**81. Weight of 1 liter of ammonia.** Arrange an apparatus as shown in Fig. 39. *A* is a 250-cc. flask containing about 100 cc. of concentrated ammonia water. *B* is a glass tube *entirely* filled with pieces of calcium oxide (unslaked lime) about the size of peas. *C* is the flask used in determining the weight of 1 liter of carbon dioxide (Fig. 30). Heat the ammonia water gently. Ammonia is evolved, is dried in passing over the lime in *B*, and collects in *C*. After *C* is filled with ammonia, proceed as in determining the weight of 1 liter of carbon dioxide. Give the results obtained.

**82. Preparation of nitric acid.** *a.* Arrange an apparatus as shown in Fig. 40. The retort *A* should have a capacity of from 100 to 150 cc. Put into the retort about 12 g. of sodium





nitrate and 10 cc. of concentrated sulfuric acid, pouring the latter through a funnel placed in the tubulus of the retort. Heat the mixture gently with a small flame. Nitric acid is generated (R), distills over, and is condensed in the test tube *B*, which should be kept cool with water. How many grams of nitric acid can be prepared from the amount of materials used?

*b.* What is the color of the acid you have prepared? What is the color of the pure acid? How do you account for the difference (R)? Try heating a little of the colored product to gentle boiling. How does this affect the color? Why?

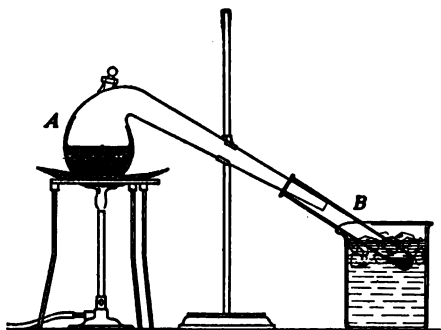


FIG. 40

**83. Action of nitric acid on metals.** *a.* Place one or two pieces of mossy tin in a test tube and add 2 or 3 cc. of the nitric acid prepared in § 82. Note the color of the evolved gas during the reaction. Does the gas in the test tube remain colored, or does the brownish color soon disappear? What is the chief gas evolved under these conditions? When the action is over, dilute to about three times the volume. The white precipitate is a hydrate of the oxide  $\text{SnO}_2$ , and for the present purpose it may be regarded as the oxide itself. Filter off the solid material, carefully collecting the filtrate. If a nitrate of tin has been formed, it should be in the filtrate, for it is soluble in water. Add an excess of ammonium hydroxide to the filtrate. Is a precipitate formed? Any tin present in the filtrate will be precipitated as the hydroxide. Verify this statement by adding a little ammonium hydroxide to a solution of tin chloride. Is tin nitrate formed by the action of concentrated nitric acid on the metal? Formulate an equation for the chief reaction, assuming that the white solid is  $\text{SnO}_2$ .



**b.** Dilute a little of the concentrated nitric acid with three times its volume of water, and pour the dilute acid upon a few pieces of copper turnings in a test tube. What is the color in the test tube as the action begins? What change in color do you notice? Hold a piece of white paper behind the test tube for a background, and compare the color of the gas at the mouth of the tube with that within the tube. How do you account for all the facts observed? What is the chief gas evolved in this reaction (R)?

**c.** Place one or two pieces of tin in a test tube, add 5 cc. of water, and then from time to time add a drop of concentrated acid. Keep shaking the solution, and continue the operation until you have added at least 1 cc. of nitric acid. Filter the solution and add ammonium hydroxide in excess. Does a precipitate form? What does this indicate? Has any noticeable quantity of red gas been evolved? From its position in the electromotive series how would you expect tin to act upon *dilute* nitric acid (R)?

**d.** Place a few pieces of zinc in a test tube and add dilute sulfuric acid until a gas is freely evolved. What is it? From time to time add a drop of concentrated nitric acid. What action would you expect to take place between nascent hydrogen and nitric acid? If ammonia is formed, what would become of it (remember what is present in the solution)? How could you test for its presence (R)? Can you detect it in the present experiment?

**e.** Give a summary of how nitric acid, both concentrated and dilute, may be expected to act upon metals *above* hydrogen in the electromotive series, and also upon those *below* hydrogen.

**84. Salts of nitric acid: nitrates.** **a.** Place a small piece of copper in an evaporating-dish (hood), add a few drops of the ordinary nitric acid, and heat gently. As soon as the copper has dissolved, carefully evaporate the solution. The flame must not touch the dish, and should be withdrawn while two or three drops of the liquid still remain in the dish. What is the composition of the residue (R)? Apply a gentle heat to the residue left in the evaporating-dish.



Account for the change in color (R). Repeat the experiment, using a piece of zinc or lead instead of copper. Are all nitrates decomposed by heat?

b. Test the solubility in water of sodium nitrate, potassium nitrate, barium nitrate, lead nitrate, and copper nitrate, using a small crystal in each case. Describe the results. What nitrates are insoluble? Try a crystal of bismuth nitrate or ferric nitrate.

c. Dissolve a crystal of sodium nitrate in 2 or 3 cc. of water in a test tube, add an equal volume of sulfuric acid, and cool. Now incline the tube and gently pour a concentrated solution of ferrous sulfate down the side of the tube, so that it floats on the heavier liquid. A brown ring soon forms where the liquids meet. Repeat the experiment, using potassium nitrate. This is a good test for nitrates. The brown ring is due to the formation of a compound of ferrous sulfate and nitric oxide, the latter being formed by the reduction of the nitric acid by the ferrous sulfate.

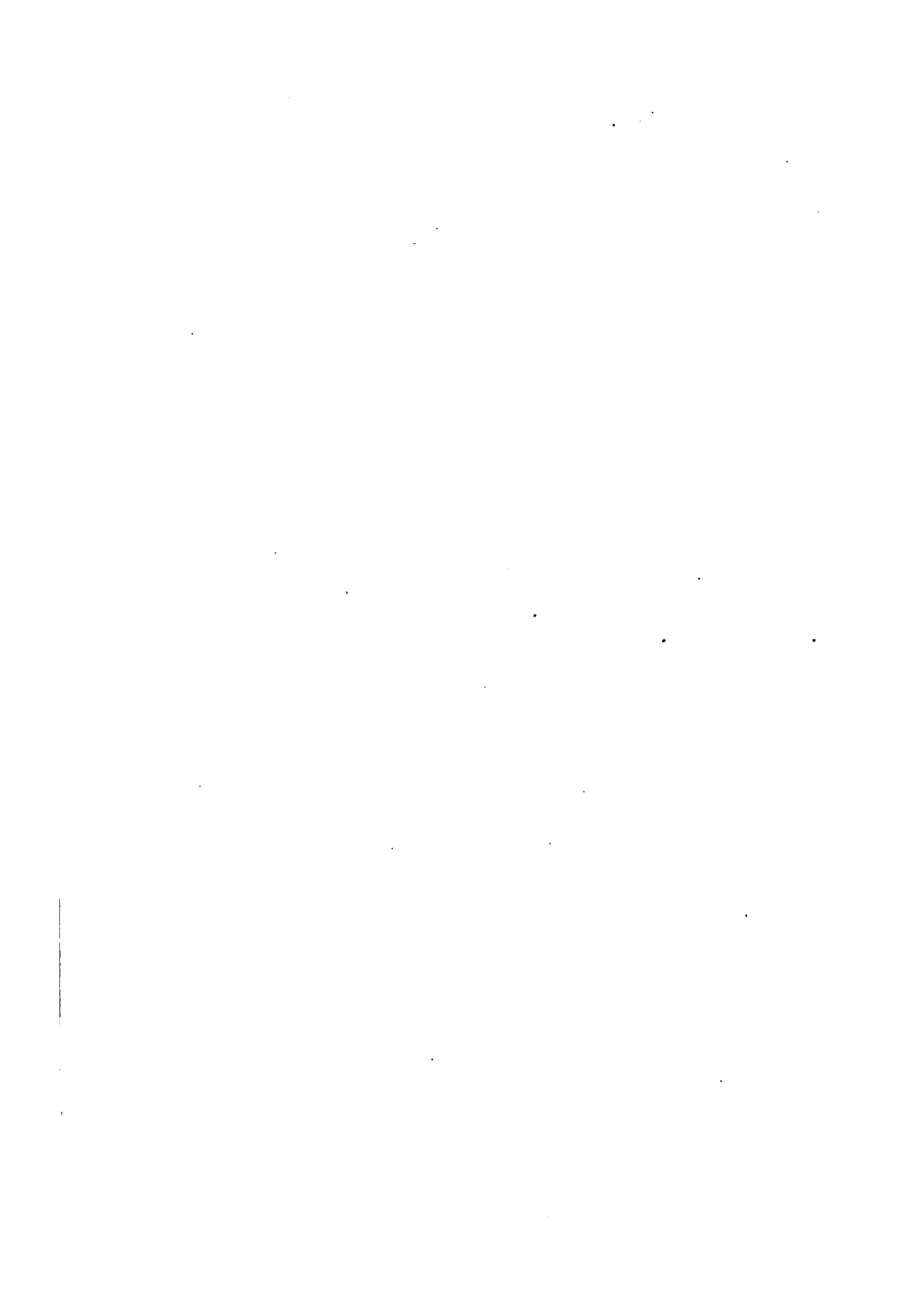
**85. Combining weights of metals from oxidation to oxides.** The following exercise will serve as an illustration of a general method. Weigh accurately a small evaporating-dish or a large crucible, place in it about 1 g. of pure copper, and again weigh. Cover the dish with a watch glass and add dilute nitric acid, a little at a time, until the metal is all dissolved. Rinse the watch glass carefully into the dish, and place the latter on the rim of a somewhat smaller beaker two thirds full of water. By boiling the water in the beaker, evaporate the solution of copper nitrate until it is nearly dry. Place the dish on a pipestem triangle and very carefully heat it with a small flame, holding the burner in the hand and using every precaution to prevent loss by spattering. As the nitrate becomes dry, gradually increase the heat until red fumes cease to be given off, finally using the full heat of the burner. Allow the dish to cool and then weigh it. What is the product? From the increase in weight calculate the ratio by weight in which copper and oxygen combine. The experiment may be varied by the use of iron, tin, or zinc in place of copper.



**86. Preparation and properties of nitrous acid.** In a hemispherical iron dish heat 10 g. of potassium nitrate until it melts and just begins to evolve bubbles; then add 25 g. of lead. Continue the heating for about twenty minutes, stirring the mixture with an iron wire or file. Note the change in color. How do you account for it (R)? When the product becomes cool add 25 cc. of water and heat until the mass is disintegrated. Filter off the residue. What is its composition? What compound is present in the filtrate? Add to the filtrate a few drops of sulfuric acid. Account for the result. Write the equation for the reaction which takes place between sulfuric acid and potassium nitrate; between sulfuric acid and potassium nitrite.

**87. Preparation and properties of some of the oxides of nitrogen.** *a. Nitrous oxide.* Put 6 or 8 g. of ammonium nitrate in the hard-glass test tube used in the preparation of oxygen. Attach a delivery tube and heat *gently*, applying no more heat than is necessary to cause a slow evolution of gas. As soon as the gas is regularly evolved collect two or three bottles of it over water. Notice the deposit of water on the sides of the test tube. What is the source of it (R)? Note the color, odor, and taste of the gas. Test it with a glowing splint. Account for the result. How can you distinguish it from oxygen? What is the common name of the gas, and for what is it used? Contrast the action of heat on ammonium nitrate and copper nitrate (R).

*b. Nitric oxide and nitrogen dioxide.* Put a few pieces of copper in your hydrogen generator (hood), just cover them with water, and add 2 or 3 cc. of nitric acid. Collect over water three bottles of the evolved gas, adding more nitric acid if necessary, and leaving the last one half filled in the pneumatic trough. Compare the color of the gas in the generator with that collected in the bottles and account for any difference. Write the equations for all the reactions involved. Uncover one of the bottles containing gas and account for the result. Test the gas in the second bottle with a burning splint. Which is the more stable, nitrous oxide or nitric





oxide? Give reasons for your answer. To the third bottle standing over water add air in small portions at a time, transferring the air to the bottle with a test tube. After the addition of each portion of air, allow the red fumes to dissolve before adding another portion. Does the volume diminish indefinitely? Why? If pure oxygen had been added instead of air, would all the gas in the bottle vanish? Can you suggest a modification of the experiment that could be used to determine the percentage of oxygen in the air?



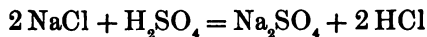
## CHAPTER XV

### EQUILIBRIUM

**88. Velocity of reactions.** *a.* To a dilute solution of sodium chloride add 1 or 2 cc. of a solution of silver nitrate. How rapidly does the reaction take place? How does it compare in rapidity with the reaction occasioned by the addition of a little barium chloride to dilute sulfuric acid? In general, reactions between freely ionized electrolytes are too rapid for measurement. Slow reactions occur when the reagents are very little ionized or when changes occur other than double decomposition between ions.

*b.* Dissolve a crystal of sodium phosphate no larger than a grain of wheat in 10 cc. of water and add 5 cc. of ammonium molybdate solution (side shelf). Does a precipitate at once form? Do you notice any change of color? Does this remain constant or increase? Set the tube aside and look at it from time to time during the laboratory period. Is the formation of a precipitate gradual or sudden? (The yellow solid has a very complex formula.)

**89. Mass action.** *a.* Make a concentrated solution of common salt by shaking 20 g. of salt with the least water that will serve to dissolve it. Add 5 cc. of concentrated sulfuric acid in small portions at a time, shaking gently after each addition (hood). The gas evolved has the formula HCl, the equation being

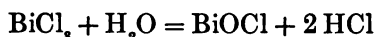


When all of the acid has been added, warm gently and set the solution aside to crystallize. (If no crystals appear by the time the solution has cooled, add a minute crystal of



sodium sulfate ( $\text{Na}_2\text{SO}_4$ .) How do the crystals differ in appearance from those of common salt? Dry a few of them and expose them to the air (R). Warm a few in a test tube. Do they contain water of hydration? Does salt? Drain the mother liquor from the crystals and pour over them about 5 cc. of concentrated hydrochloric acid. What members of the above equation have you brought together? Do you note any change in the crystals? Filter some of them and dry them on filter paper. How can you prove that they are common salt? Complete the equation. How does it compare with the one written above? Why has the reaction been reversed?

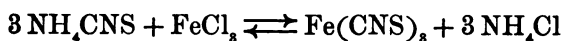
*b.* Place a few crystals of bismuth chloride ( $\text{BiCl}_3$ ) in a beaker and add about 1 cc. of water. What change do you notice? The equation is



Under what conditions would you expect the reaction to be reversed? Add concentrated hydrochloric acid, a drop at a time, with constant stirring. What is observed (R)? When the solution is complete, add water, a little at a time, until the precipitate again appears. Can you again reverse the reaction? How many times can you repeat the process?

**90. Equilibrium in solution.** *a.* Dissolve about 1 g. of cupric bromide ( $\text{CuBr}_2$ ) in the least possible volume of water. What is the color of the solution? Add water, a small quantity at a time, until the solution becomes a clear green. Pour off half the solution and dilute it further. What is the final color? How can you account for the intermediate green? To the remaining half of the green solution add crystals of potassium bromide or sodium bromide. What change in color is noticed? How do you explain this?

*b.* Obtain about 5 cc. of a solution of ammonium sulfo-cyanate ( $\text{NH}_4\text{CNS}$ ) and of ferric chloride ( $\text{FeCl}_3$ ) from the side shelf. Pour 1 cc. of each of these solutions into 50 cc. of water. An equilibrium results as follows:





The compound  $\text{Fe}(\text{CNS})_3$  is deep red, and even in very dilute solution has a noticeable pink color. Half fill four test tubes with the solution, and reserve one tube for comparison. To the second tube add 1 or 2 cc. of the solution of ammonium sulfocyanate. What change in color results? Note the equation and explain. To the third tube add 1 or 2 cc. of the solution of ferric chloride. Explain the change. To the fourth tube add 2 or 3 g. of solid ammonium chloride. Explain the change.





## CHAPTER XVI

### SULFUR AND ITS COMPOUNDS

**91. Properties of sulfur.** *a.* Examine the physical properties of a piece of brimstone. Pour 2 or 3 cc. of carbon disulfide over a little powdered brimstone in a test tube (*keep away from flame*). Cover the mouth of the tube with the thumb and shake the contents gently until the sulfur is dissolved. Pour the clear solution into an evaporating-dish, cover it loosely with a filter paper, and set it aside in the hood. The carbon disulfide soon evaporates, the sulfur being deposited in crystals. Examine these with a magnifying-glass.

*b.* Half fill a test tube with powdered brimstone and apply heat enough to just melt it. Note the properties of the liquid. Pour a little of the liquid into a beaker of water, dry the product on filter paper, and test its solubility in carbon disulfide. Now apply a stronger heat and observe that the liquid becomes darker and at a certain temperature ( $200^{\circ}$ – $250^{\circ}$ ) is so thick that the tube may be inverted without spilling it. Finally, increase the heat until the sulfur boils ( $445^{\circ}$ ), and pour the boiling liquid into a beaker of cold water. Examine the product. What name is given to this form of sulfur? Dry a small piece on filter paper and test its solubility in carbon disulfide. Expose it to the air for a day or two. Have its properties remained unchanged?

*c.* Fill a porcelain crucible with powdered brimstone and apply a very gentle heat until the sulfur is just melted. Withdraw the flame and watch the liquid carefully as it cools. Crystals soon begin to form on the surface, rapidly growing from the circumference toward the center. Before they reach the center quickly pour the remaining liquid into



a dish and examine the crystals adhering to the sides of the crucible. Compare them with those formed in *a*. In how many forms have you obtained the sulfur? Which is the stable form?

*d*. Burn a small piece of sulfur. What is the product of the combustion? Note the odor of it. Heat to boiling a little sulfur in the test tube used in *b* and drop a small strip of bright copper foil into the boiling liquid. Is there any visible evidence of a chemical reaction? What is formed? What is the product formed in § 11, *b*?

**92. Transition point.** The transition of monoclinic sulfur into rhombic is too slow to be observed readily. The following is a more striking experiment: Obtain a piece of thin-walled glass tubing about 15 cm. long and 5 mm. in diameter. Seal one end in the Bunsen flame, blowing the seal into rounded form like a test tube. Obtain about half a gram of cupromercuric iodide (side shelf) and put it into this test tube. Half fill a beaker with water, place it on a wire gauze on a ring stand, and suspend a thermometer in such a way that the bulb dips into the water. Heat the water until the temperature reaches about 60°, then turn the flame down so that the rise in temperature is very slow. Use the test tube containing the scarlet powder as a stirring-rod and watch for a change in color. What change occurs? At what temperature? Withdraw the flame, allowing the temperature to fall slowly. At what point does the reverse change occur? Repeat the experiment to ascertain within what limits of temperature the change occurs. Return the powder to the stock bottle. Cupromercuric iodide is sometimes used as a paint for bearings in machinery, since its change in color indicates that the bearing is becoming heated.

**NOTE.** If desired, the student may himself prepare the cupromercuric iodide as follows: Dissolve a few crystals of mercuric chloride in 2 or 3 cc. of hot water in one test tube, a few crystals of potassium iodide in another, about the same weight of copper sulfate in a third, and a similar quantity of sodium sulfite in a fourth. Add the potassium iodide, drop by drop, to the mercuric chloride until the bright scarlet



precipitate just redissolves. To the solution of sodium sulfite add a little moderately dilute sulfuric acid, and pour this solution into the one just prepared; then quickly add the copper sulfate. Collect the red cupromercuric iodide on a filter paper, wash it once or twice with cold water, and dry the powder on sheets of filter paper.

**93. Hydrogen sulfide.** *a.* (Hood.) Attach a delivery tube to the hydrogen generator, as represented in Fig. 41. Put into the generator a few pieces of iron sulfide ( $\text{FeS}$ ) and insert the stopper. Before generating the gas have at hand a dry bottle in which to collect some of it, a short piece of glass tubing of size to fit the rubber connection *A* and drawn to a jet at one end, also test tubes about one fifth full of solutions of each of the following compounds: copper sulfate, zinc sulfate, cadmium sulfate, magnesium sulfate, sodium chloride. Now pour a few drops of a dilute solution of sulfuric acid (1 part acid to 7 parts of water by volume) through the funnel tube of the generator, adding more from time to time, if

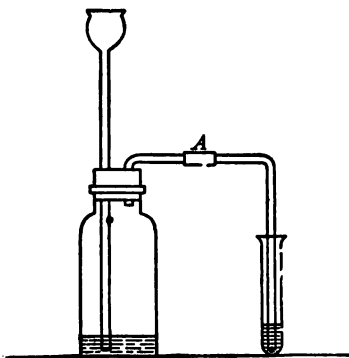


FIG. 41

necessary, to produce a gentle evolution of the gas (R). Be careful not to inhale the gas, as it is poisonous.

*b.* Expose a piece of moist litmus paper to the gas. What is its reaction? Dip a piece of filter paper into a solution of lead acetate (side shelf) and expose it to the gas (R).

*c.* Collect a bottle of the gas by downward displacement, allowing the gas to flow until it is ignited by a flame held at the mouth of the bottle (R). Account for the deposit on the sides of the bottle. Connect the glass jet with the delivery tube at *A* and ignite the gas at the tip. Note the appearance of the resulting flame. What are the products of the combustion? Hold a cold porcelain dish in the flame. What is deposited on the dish? Why?



d. Cause the gas to bubble for a few seconds through each of the solutions in the test tubes (Fig. 41), noting the color of the precipitates obtained. What is the composition of each (R)? In which cases does no precipitate form? Why is this?

e. Pass a little of the gas into some water in a beaker. How does the solution compare with natural sulfur waters? Note the odor of the solution. Pour a little of it into a test tube and boil for a short time. Does the odor disappear? Can the gas be expelled from the water by boiling? Drop a silver coin into the solution, and account for the results.

f. Pass the gas into a solution of zinc sulfate as long as a precipitate continues to form. If the action is a completed one, will any zinc sulfate remain in the solution? Filter from the zinc sulfide and add a little ammonium hydroxide to the filtrate. How do you account for the additional precipitate?

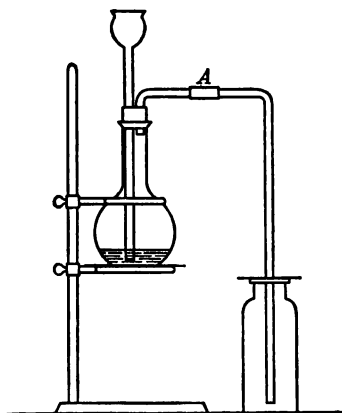


FIG. 42

— **94. Sulfur dioxide.** Place about 10 g. of copper turnings or small pieces of sheet copper in a generator arranged as in Fig. 42 (hood). Add about 25 cc. of concentrated sulfuric acid and apply a gentle heat. As soon as the action begins (R), lower the flame, regulating it so as to obtain a uniform evolution of the gas. Collect two bottles of the gas by downward displacement; then cause it to bubble through 25 cc. of water as long as any is dissolved. Do not allow the delivery tube to remain in the solution after the gas ceases to flow, or the solution will run back into the flask (why?). By what other method has this gas been prepared in a previous exercise? Invert one of the bottles of the gas so that the mouth of it is under water, and examine the contents after several minutes. In the second bottle place some moist paper containing writing





in ink, some paper with print upon it, or some moistened flowers or colored cloth. Are the colors affected? Contrast the action of hot concentrated sulfuric acid upon copper with that of dilute sulfuric acid on zinc or iron (R).

**95. Sulfurous acid.** *a.* Test the saturated aqueous solution of sulfur dioxide with blue litmus. Is the gas combined with the water or simply dissolved in it? Give reason for your answer. Boil a little of the solution vigorously for a time. Does the odor persist? How do you account for this? To some of the concentrated solution add a solution of sodium hydroxide, drop by drop, until it becomes neutral (R), and then evaporate just to dryness. What is the residue? Moisten it with 2 or 3 drops of sulfuric acid and note the odor of the gas evolved. What is it? How could you test for a sulfite?

*b.* To 5 cc. of the solution of sulfur dioxide add a drop or two of barium chloride. Does a precipitate form? Is it soluble in hydrochloric acid? Repeat the experiment, first adding a little concentrated nitric acid, and boiling before adding the barium chloride. Do you note any differences? How do you explain them?

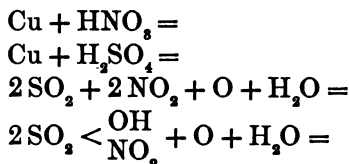
*c.* Place about 10 cc. of water in a bottle and saturate it with sulfur dioxide; then pass hydrogen sulfide into the solution. What is the precipitate (R)? Can you filter it off? What is this form called?

**96. Weight of 1 liter of sulfur dioxide.** Determine the weight of 1 liter of sulfur dioxide according to the method suggested for carbon dioxide (§ 52), making the necessary alterations in the method. Will it be necessary to dry the gas? Why?

**97. Sulfuric acid.** *a. Usual laboratory method* (hood). Arrange an apparatus according to Fig. 43, in which *B* represents a wide-mouthed bottle of about 1 liter capacity. The bottle should be rinsed out with water but *not* dried. By the action of *concentrated* nitric acid on copper, generate nitrogen dioxide in *C* until *B* is filled with red fumes. Now pass in sulfur dioxide from *A* until the red fumes entering from *C* are completely decolorized; then replace *A* with a flask containing



a little water, boil the water, and conduct the steam into *B* until 5 or 10 cc. of liquid has been collected. Pour this into a beaker and save it for further experiments. Complete the following equations:



*b.* Add 3 drops of sulfuric acid to 5 cc. of water in a test tube. To this add a few drops of a solution of barium chloride. Note that a precipitate forms (R). Now add 3 or 4 drops of hydrochloric acid. Does the precipitate dissolve? The formation with barium chloride of a precipitate *which is insoluble in hydrochloric acid* constitutes a good test for sulfuric acid.

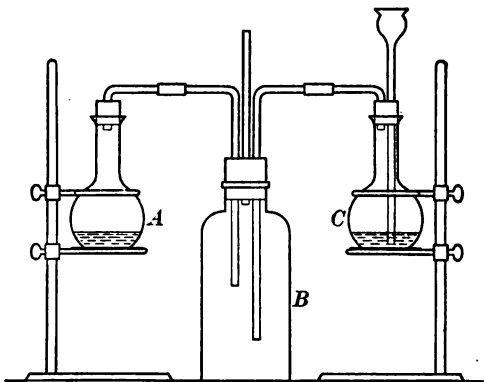


FIG. 43

Now apply this test to the liquid taken from the bottle *B* (Fig. 43). Note the results.

*c.* Recall the action of dilute sulfuric acid on zinc (R). Try the action of the concentrated acid on zinc, applying a gentle heat, if necessary, to start the reaction. What gas is now evolved? Explain. Write in steps the equations for the reactions which take place when copper is acted on by nitric acid and hot concentrated sulfuric acid respectively, pointing out the similarity in the action of the two acids. Gently heat a small bit of charcoal with 2 or 3 cc. of concentrated sulfuric acid. What gas is evolved (R)?



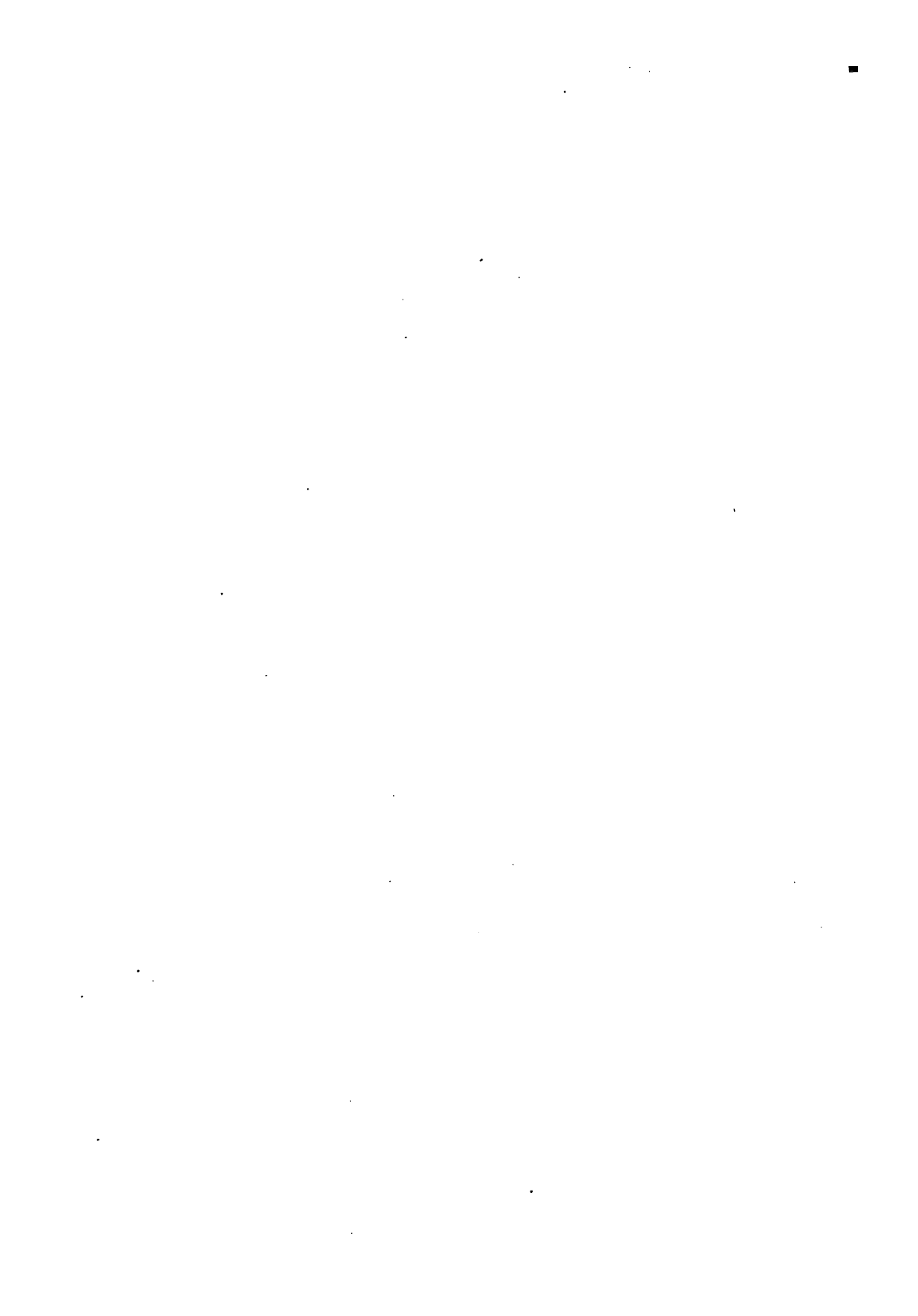
*d.* Put a drop of concentrated sulfuric acid on a splint and gently warm it above a flame. Pour a few drops on 0.5 g. of sugar in a test tube. Examine after a few minutes. Account for the results.

*e.* Why is sulfuric acid used in the preparation of other acids?

**98. Salts of sulfuric acid : sulfates.** *a.* Obtain a crystal of each of five or six different sulfates, dissolve them in water, and test each solution with litmus paper. Why do some of the sulfates have an acid reaction? Try to dissolve a crystal of sulfate of antimony, of bismuth, or of mercury in pure water. What do you observe? Explain.

*b.* Dissolve in a little water a crystal of each of the following sulfates: sodium sulfate, magnesium sulfate, copper sulfate. Apply to each the barium chloride test for sulfuric acid (R). Can sulfates be detected by this method? Dissolve a crystal of sodium sulfite in water and apply the same test. How can you distinguish between sulfates and sulfites? (Sulfites very often contain more or less of the corresponding sulfates, owing to the absorption of oxygen from the atmosphere.)

*c.* Is sulfuric acid monobasic or dibasic? Explain these terms. With due care pour about 10 cc. of concentrated sulfuric acid into 100 cc. of water, and divide the solution into two equal parts. Carefully neutralize the one part with sodium hydroxide and evaporate the solution to about 25 cc. Set aside for a day to obtain crystals. What are they? Divide the second part into two equal portions. Neutralize the one part as before; then add the second portion. What should be in solution? Evaporate as in the other case and set aside to crystallize. Compare the two kinds of crystals in appearance. Heat a few of each kind in a dry test tube. What difference do you note?



## CHAPTER XVII

### THE CHLORINE FAMILY

**NOTE.** The experiments on chlorine, hydrogen chloride, and hydrochloric acid have been given in Chapter X. The student should review these in connection with the present chapter.

**99. Hydrogen fluoride; hydrofluoric acid.** (*The gas is very corrosive and must not be inhaled; its solution must not be brought in contact with the skin.*) Warm gently over a small, luminous flame a glass plate on which some pieces of paraffin have been placed. When the paraffin is melted, tilt the plate about so as to cover it with a uniform layer of the wax. When the plate becomes cold, scratch your name *through* the wax with a pin or other sharp point. Place in a lead dish 3 g. of powdered fluorite and add sufficient sulfuric acid to make a paste of it. Cover the dish tightly with the waxed side of the glass plate and set it in the hood for an hour; then remove the paraffin and examine the glass. Write the equations for all the reactions involved. In what kind of bottles is hydrofluoric acid stored?

**100. Salts of hydrochloric acid: chlorides.** Place in separate test tubes a few drops of a solution of each of the following compounds: sodium chloride, potassium chloride, calcium chloride. Add to each 1 or 2 drops of a solution of silver nitrate. Explain the results (R). Add a few drops of ammonium hydroxide to each test tube, and note the result. Now add nitric acid, drop by drop, to each test tube until the liquid is acid, and note the result. The formation with silver nitrate of a white precipitate which is soluble in ammonium hydroxide and insoluble in nitric acid serves as a good test for chlorides. Will this test also serve to detect the presence of hydrochloric acid?





**101. Weight of 1 liter of hydrogen chloride.** The weight of 1 liter of hydrogen chloride may be determined by the method employed in determining the weight of 1 liter of carbon dioxide (§ 52). Prepare the gas as described in § 64. The gas should be bubbled through concentrated sulfuric acid in bottle *B* (Fig. 34), in order to dry it.

**102. Preparation and properties of bromine.** (*The vapor of bromine must not be inhaled. Perform the experiments in a hood.*) *a.* Use the apparatus employed in the preparation of nitric acid (Fig. 40). Put into the retort a mixture of 2 g. of potassium bromide or sodium bromide and 4 g. of manganese dioxide, and add to this through a funnel a cold dilute solution of sulfuric acid, prepared by slowly adding 5 cc. of sulfuric acid to 10 cc. of water. Shake the retort so as to mix the contents thoroughly. The test-tube receiver should contain sufficient water to allow the end of the retort to dip just below its surface. Now heat the retort gently. The bromine is liberated and distills over (*R*). Continue the heating until all the bromine has been collected.

*b.* Note the properties of the bromine collected in the bottom of the receiver. Has the water dissolved any of it? What property is implied in the name of the element? Test the bleaching conduct of the aqueous solution. How does it compare with chlorine as a bleaching agent? Try the effect of a little of the bromine water on starch solution.

*c.* Pour 2 or 3 cc. of the solution of bromine into each of two test tubes. To the one add an equal volume of carbon disulfide and shake it vigorously. What is the distribution of the bromine between the two solvents? To the other test tube add carbon tetrachloride or chloroform and compare the results with those obtained with carbon disulfide.

**103. Hydrobromic acid and the bromides.** *a.* Add a few drops of sulfuric acid to some crystals of potassium bromide. Note the formation of white fumes, and also of a reddish-brown vapor. Give the composition of each and account for its formation. (Recall that concentrated sulfuric acid is an oxidizing agent and hydrobromic acid is a reducing agent.)



**b.** Dissolve in water a crystal of potassium bromide or of sodium bromide and apply the silver nitrate test for chlorides (§ 100). Describe the results (R).

**c.** To a solution of a crystal of a bromide add a little chlorine water. How do you account for the change in color? Shake the solution with 2 or 3 cc. of carbon tetrachloride. Does this make the test more delicate?

**104. Preparation and properties of iodine.** In a large test tube place a mixture of 2 g. of potassium iodide and 4 g. of manganese dioxide. Pour over this mixture 5 cc. of sulfuric acid, place an empty funnel in the tube to serve as a loose stopper, and apply a gentle heat (R). Note the vapor of the iodine and the grayish-black crystals which are soon deposited on the sides of the test tube. What property does the name of the element suggest? Half fill two test tubes with starch solution. Add to the first a few drops of a solution of iodine prepared by shaking a small crystal in water. To the second add a few drops of an aqueous solution of potassium iodide. Note the results. Now add to the second tube a little chlorine water (R). Does the chlorine water alone change the starch? Explain the results. Add about 2 cc. of carbon tetrachloride, and shake the mixture. In which solvent is the iodine more soluble? Dissolve a crystal of iodine in alcohol. This solution is called tincture of iodine.

**105. Hydriodic acid and the iodides.** **a.** Repeat the experiment of § 103, **a** and **b**, substituting potassium iodide for the bromide. Note and explain the results.

**b.** Grind to a fine powder two or three crystals of iodine in a mortar and add a little water. Transfer the resulting mixture to a test tube, adding sufficient water to nearly fill the tube. Now pass a slow current of hydrogen sulfide through the liquid until the iodine all disappears. Filter off the white solid (explain) and boil the filtrate until the hydrogen sulfide is expelled (to test for the presence of hydrogen sulfide, hold in the vapor a strip of paper moistened with a solution of a salt of lead; if the acid is present in the vapor, the strip turns dark, owing to the formation of black lead



sulfide). Add a few drops of a solution of silver nitrate to the resulting liquid. Explain.

**106. To distinguish between chlorides, bromides, and iodides.** Can you distinguish between chlorides, bromides, and iodides by means of the silver nitrate test? Try the following test and describe the results: Place in separate test tubes about 5 cc. of solutions of each of the following compounds: sodium chloride, sodium bromide, sodium iodide. Add to each about 1 cc. of carbon disulfide and a few drops of chlorine water. Shake the solutions in each test tube so as to mix them *thoroughly*. If both a bromide and an iodide were present, could you detect the bromide? Try the addition of more chlorine water to the iodide. What becomes of the iodine (remember that the chlorine is a good oxidizing agent in the presence of water)?

**107. Hypochlorites and chlorates.** *a.* Dissolve about 6 g. of solid potassium hydroxide in 18 cc. of cold water, and pour half of the solution into a good-sized test tube, reserving the other half for § 107, *c.* Fit up a small chlorine generator and pass a rapid current of chlorine into the solution, taking care to keep the test tube cold by immersing it in cold water. When no more chlorine is absorbed (ten to fifteen minutes) and the solution is no longer strongly soapy to the touch, withdraw the test tube and replace it by the one containing the other half of the solution of potassium hydroxide. This test tube should be previously heated until it is moderately warm, and it should not be cooled while the chlorine is passed in. Continue the operation until the solution is saturated.

*b.* Pour the contents of the first test tube into a beaker. To 1 cc. of the solution add an excess of nitric acid and then a few drops of silver nitrate. What inference do you draw? Place a piece of brightly colored cloth in the remainder of the solution. Is it bleached? Now add an excess of dilute sulfuric acid. Is the bleaching more rapid than before? How does the sulfuric acid assist in the bleaching?

*c.* Pour the contents of the second test tube into a beaker and set aside to cool, if necessary, till the next period. Filter



off the crystals and wash them with a little cold water, then dry them on filter paper. Dissolve a crystal in water and test with silver nitrate (R). Is the substance a chloride? Heat the crystals in a test tube and test for oxygen. Dissolve the residue in water and test for the presence of a chloride. Add a little silver nitrate to the solution from which the crystals were obtained. Is a chloride present? Write the equations for the action of chlorine on potassium hydroxide.

**108. Bleaching-powder.** Arrange an apparatus according to Fig. 44. In this apparatus *A* represents a 250-cc. flask in which chlorine is generated according to § 62, *d*, using 15 g. of manganese dioxide and 75 cc. of hydrochloric acid. The chlorine must be evolved *slowly*, and so only a *very gentle* heat is applied to the flask. The gas is conducted through a little water in *B*,

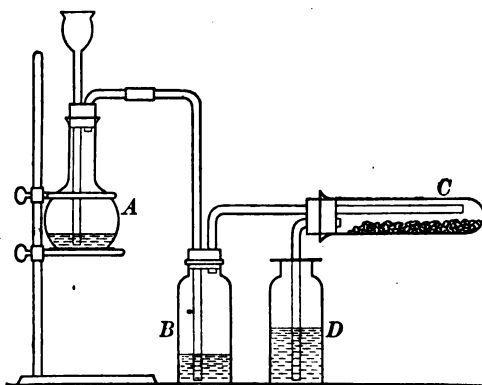
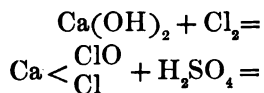


FIG. 44

then into the tube *C*, which is half filled with slaked lime. The bottle *D* contains a solution of sodium hydroxide. When all the chlorine has passed over from *A*, disconnect the apparatus and transfer the contents of the tube *C* to a beaker (200-cc. to 300-cc.) and pour over it a little sulfuric acid diluted with an equal volume of water. Hang a moist strip of red calico in the beaker and cover the beaker with a glass plate. Note the results. Complete the following equations:



What is the function of the liquids in *B* and *D*?





## CHAPTER XVIII

### SOME COMPOUNDS OF CARBON

NOTE. Students will select either § 109 or § 110, according to whether formic acid or oxalic acid is to be used in the preparation of carbon monoxide. *It must be kept in mind that carbon monoxide is a very poisonous gas. It must not be allowed to escape in the laboratory.*

#### 109. Carbon monoxide: its preparation from formic acid.

a. Arrange an apparatus according to Fig. 45. Remove the stopper from the flask *B* (Fig. 45), pour in 15 cc. of sulfuric acid and connect the apparatus as shown in the figure. Close the clamp *C* and partially fill the funnel *A* with formic acid (50 per cent acid). Now open the clamp carefully so that the formic acid will enter the flask, a drop at a time. Allow

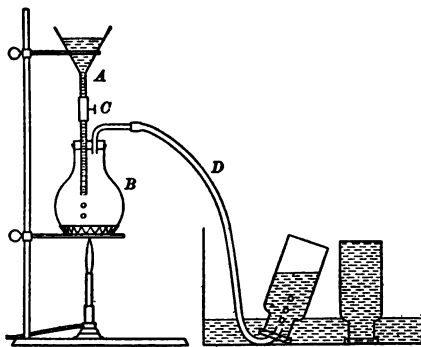


FIG. 45

8 or 10 drops to flow in; then close the clamp. If the reaction does not begin (as indicated by absence of effervescence of the liquid in the flask and escape of gas through the exit tube), heat the flask very gently until the reaction starts; then open the clamp again and admit the formic acid, a drop at a time, so as to secure a regular flow of gas from the flask. If necessary, add more formic acid to the funnel so as to keep it partially filled (?). Collect three bottles of the gas as shown in the figure. Close the clamp so as to stop further



generation of gas. Slip the glass plates over the mouths of the bottles and remove the bottles from the trough. In the first bottle filled, is the gas pure carbon monoxide? Remove the glass cover and test it with a flame (?). Repeat with the second bottle filled (?). Slip the glass plate from the third bottle just far enough to pour into the bottle 5 cc. of clear limewater; then quickly replace the glass plate and, holding it firmly against the mouth of the bottle, shake the contents of the bottle. Note any change in the appearance of the limewater. Now tip the bottle as far as possible without spilling the limewater; remove the glass plate and quickly ignite the gas, holding the bottle in this position so that at least a portion of the combustion product may be retained in the bottle. When the flame dies out, at once cover the mouth of the bottle with the glass plate and shake the contents. Note the results.

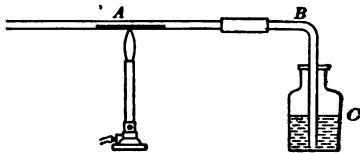


FIG. 46

b. Introduce into the tube *A* (Fig. 46), a small amount of copper oxide and arrange the apparatus as shown in the figure. Now connect the exit tube *D* (Fig. 45) with the tube *A* (Fig. 46). Heat the copper oxide gently; at the same time pass a slow current of carbon monoxide through the tube, generating the gas as described in *a*. Continue until the limewater and copper oxide both have visibly changed. Describe the results and write the equations for all the reactions involved.

#### 110. Carbon monoxide: its preparation from oxalic acid.

a. Arrange an apparatus according to Fig. 47. The flask *A* should have a capacity of from 100 to 150 cc. The bottle *B* contains a solution of sodium hydroxide, and *C* and *D* contain solutions of limewater. The hard-glass tube *E* (12 to 15 cm. in length) contains, near its middle, a layer of 1 or 2 g. of black copper oxide. The whole apparatus must be air-tight. After the apparatus has been approved by the instructor, put 7 or 8 g. of oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ) into the flask *A*,



and pour over this 25 cc. of concentrated sulfuric acid. Stopper the flask tightly and apply a very gentle heat, at the same time heating the copper oxide in *E*. Regulate the heat so as to cause the evolved gas to bubble *slowly* through *B* and *C*. Collect over water in *F* any gas escaping from *D*. What evidences have you that the copper oxide is reduced?

*b.* Disconnect the bottle *B* and attach a rubber delivery tube in its place. Collect by displacement of water a test tube full of the gas evolved in *A*. Close the mouth of the tube

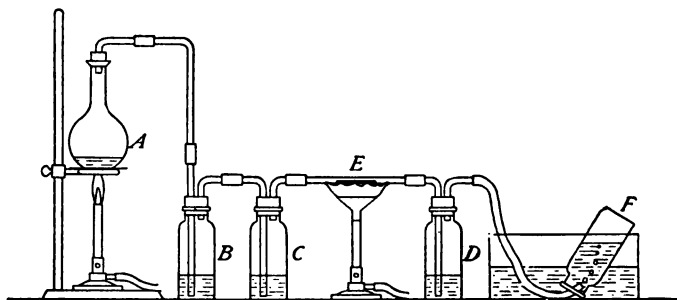


FIG. 47

with the thumb and invert it in a beaker containing a solution of sodium hydroxide. What proportion of the gas is absorbed? What is it? What gas remains?

*c.* Withdraw the heat from *A*, remove the stoppers from *A*, *B*, and *C*, and test with a burning splint the gas inclosed in each. Account for the results. Also test with a burning splint the gas which escaped from *D*, and account for the result. Why is the gas passed through a solution of sodium hydroxide? Write the equations for the reactions which take place in each of the containers *A*, *B*, *C*, *D*.

**111. Carbonic acid and its salts.** *a.* Generate carbon dioxide as in § 51. Wash the gas by bubbling it through water, and then pass it through pure water in a beaker until the gas is no longer absorbed. Test the solution with litmus. How does the taste of the solution compare with that of water?



**b.** Give the formula and properties of the acid of which the carbonates are salts. Try the action of hydrochloric acid on a small amount (about 1 g.) of each of the following carbonates: sodium carbonate, potassium carbonate, magnesium carbonate (R). How can you detect the presence of carbonates? Is limestone a carbonate? Is the action of sulfuric and nitric acids on carbonates similar to that of hydrochloric acid (R)? Why is carbonic acid so readily liberated from carbonates?

**c.** Pass a current of carbon dioxide through 10 cc. of the laboratory solution of sodium hydroxide (1 part of sodium hydroxide to 10 parts of water). Does the solution remain clear? When no more of the gas is absorbed, evaporate the solution to dryness and test the residue for carbonates. What is the residue (R)?

**d.** In what respects does carbon dioxide resemble sulfur dioxide?

**e.** Half fill a small beaker with limewater and pass carbon dioxide through the liquid (R). Continue until the precipitate which at first forms is dissolved (R), and then divide the solution into two parts. To the one add a little clear limewater (R); to the other apply heat until it boils rapidly (R). Account for the fact that carbon dioxide will cause a precipitate in a solution of calcium hydroxide but not in a solution of sodium hydroxide.

**112. Oxidation of urea.** Prepare about 100 cc. of a solution of sodium hypochlorite by passing chlorine into a *cold* solution of sodium hydroxide (§ 107) until the chlorine is no longer readily absorbed, thus leaving some sodium hydroxide present in solution with the sodium hypochlorite (R). Fill a test tube with the solution and invert it in a small evaporating dish partially filled with the same solution. Now, by means of a medicine dropper, the small end of which is curved slightly, introduce into the solution in the test tube a few drops of a solution of urea (R).





## CHAPTER XIX

### THE LAW OF GAY-LUSSAC

NOTE. Before performing the experiment, read the directions carefully and report to the instructor for quiz on the methods.

113. Determination of the volume of hydrogen obtained from a known volume of hydrogen chloride. Prepare some sodium amalgam as follows: Pour about 10 cc. of mercury into an evaporating-dish and heat slightly (hood). Add, one at a time, five pieces of sodium, each as large as a pea. If each bit of the sodium does not combine with the mercury on coming in contact with it, start the combination by pushing the sodium under the surface of the mercury by means of a long glass rod. After the sodium has all been added, pour the resulting amalgam into a small bottle and stopper it.

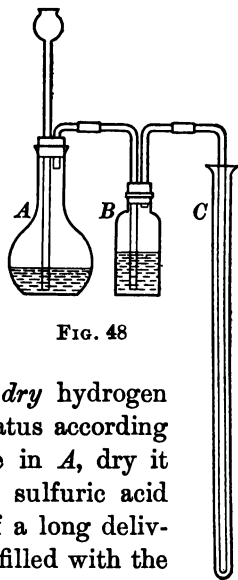


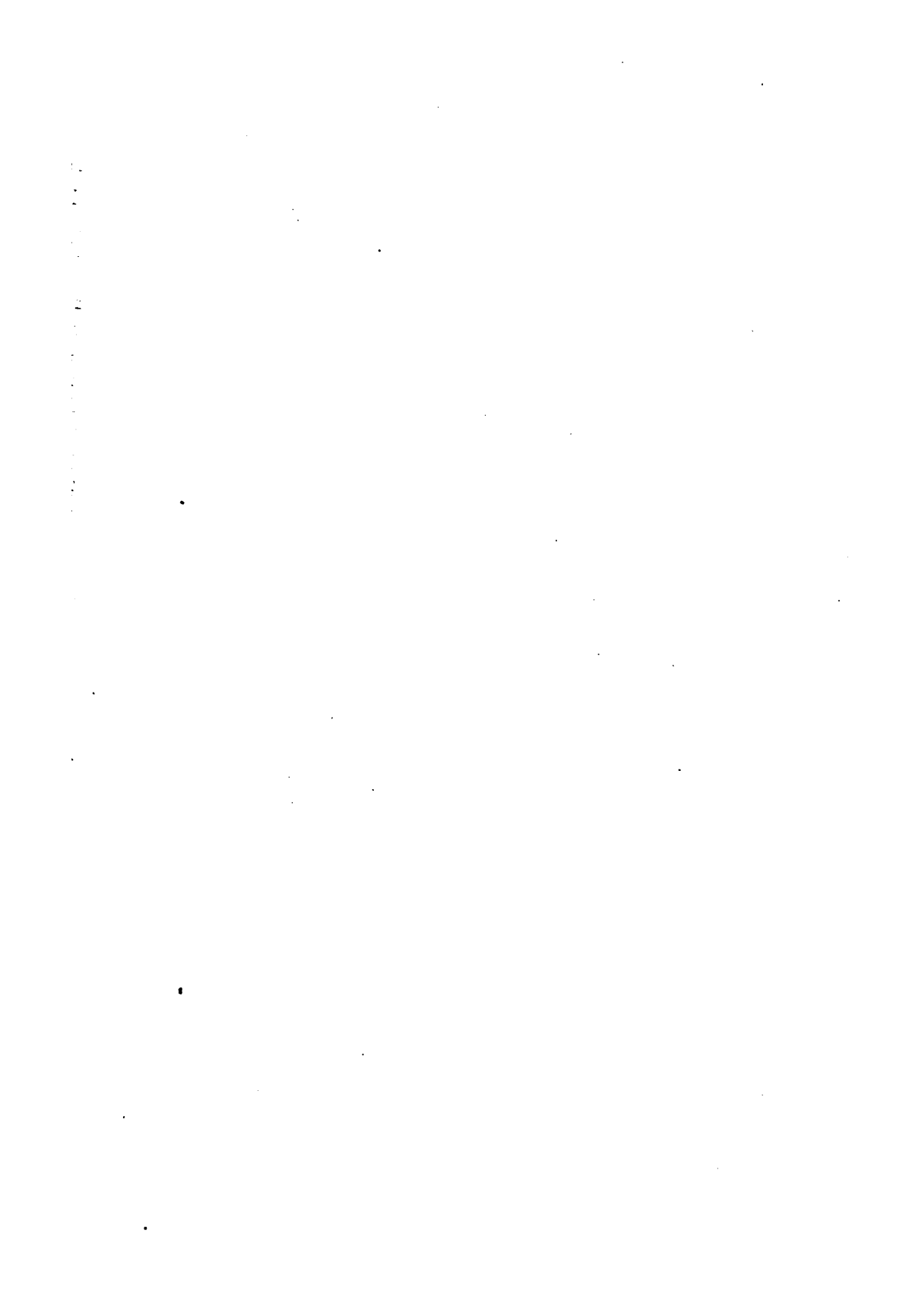
FIG. 48

Obtain from the storeroom a tube about 50 cm. in length and 15 mm. in diameter (Fig. 48, C), and fill it with *dry* hydrogen chloride. To do this, arrange an apparatus according to Fig. 48. Generate hydrogen chloride in A, dry it by passing it through the concentrated sulfuric acid in B, and conduct it into C by means of a long delivery tube. When the tube C is *completely* filled with the gas, *slowly* withdraw it from the delivery tube, pour in the sodium amalgam prepared above, and *at once* close the mouth of the tube firmly with the thumb, slightly moistened. Now shake the tube so as to bring the gas into contact with



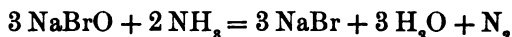
the amalgam. Finally, invert the tube in a vessel of water and remove the thumb. The water rises in the tube. Determine the nature and the exact volume of the resulting gas. Draw conclusions with reference to the composition of hydrogen chloride.

114. Determination of the relative volumes of nitrogen and hydrogen obtained by the decomposition of ammonia. Use the apparatus shown in Fig. 32, except that the glass tube *C* used in § 113 is substituted for the test tube. Clean and dry the tube *C* and fill it with dry chlorine gas (hood). The chlorine is generated in *A* (Fig. 48), is dried by passing it through the sulfuric acid in *B*, and is conducted into *C* by means of the long delivery tube. While the tube is being filled, pour into the funnel *A* (Fig. 32) a few cubic centimeters of a concentrated solution of ammonium hydroxide. Open the clamp *B* until the rubber tube and the glass nozzle are completely filled with the solution; then close the clamp. When the tube *C* is completely filled with chlorine, slowly withdraw it from the delivery tube and at once connect it with *B* (Fig. 32) by means of the cork (the cork must be firmly inserted so as to make the connection air-tight). Now allow the ammonium hydroxide solution to enter the tube, a drop at a time. The chlorine combines with the hydrogen of the ammonia and liberates the nitrogen (R). When the addition of the hydroxide solution no longer causes any action, pour into the funnel some dilute sulfuric acid and admit this to the tube so as to combine with the excess of ammonia. The tube should be shaken so as to mix the contents thoroughly. When the sulfuric acid no longer causes any action, fill the funnel with water and open the screw clamp (care must be taken that no air is admitted to the tube with the liquids). When no more water will enter the tube, close the screw clamp, remove the funnel, and invert the tube in a trough of water. Remove the cork, adjust the pressure, and determine the exact volume and the nature of the remaining gas. What volume of hydrogen was necessary to combine with the volume of chlorine taken? What was the source of this



hydrogen? What became of the liberated nitrogen? How does the volume of nitrogen liberated compare with the volume of hydrogen used in combining with the chlorine? What are the relative volumes of hydrogen and nitrogen set free by the decomposition of ammonia?

**115. Determination of the volume of nitrogen obtained from a known volume of ammonia.** *a.* Use the same apparatus as in § 114. Clean and dry the tube *C* and fill it with dry ammonia gas by upward displacement. The gas is most conveniently generated by warming a concentrated solution of ammonium hydroxide. It is dried by passing it through a tube filled with small pieces of lime, and is then conducted through the long delivery tube into *C*. While the tube is being filled, pour into the funnel (Fig. 32) a solution of sodium hypobromite (side shelf). Open the clamp until the rubber tube and the nozzle are completely filled with the solution, then close the clamp. When the tube *C* is completely filled with ammonia, slowly withdraw the delivery tube and quickly connect (air-tight) the tube with *B* (Fig. 32) by means of the cork. Now admit the hypobromite solution slowly as long as any action takes place. The following equation represents the reaction involved:



When the action has ceased, water is admitted. When no more water will enter the tube, remove the funnel, invert the tube in a trough of water, and remove the cork. Adjust the pressure and determine the volume and nature of the remaining gas. How does the volume of nitrogen obtained from the ammonia compare with the volume of the ammonia?

*b.* From the results obtained in §§ 114 and 115, *a*, state the relations between the volumes of hydrogen and nitrogen which combine to form ammonia, also their relation to the volume of ammonia formed.

**116. Comparison of results obtained.** Recall (lecture experiment) the relation between the volumes of hydrogen and oxygen which combine to form water, also the volume of

[illegible]

water vapor formed. Recall also the relation between the volumes of hydrogen and chlorine which combine to form hydrogen chloride, and the relation each holds to the volume of hydrogen chloride formed. Represent graphically the proportion by volume in which the two elements in each of the following pairs of elements combine, and also the relative volume of the compound formed in each case by their union: hydrogen and chlorine; hydrogen and oxygen; hydrogen and nitrogen. State all of these facts in the form of a generalization. What is this generalization called?





## CHAPTER XX

### COMBINING WEIGHTS AND MOLECULAR WEIGHTS

**117. The combining weight of zinc.** From your results obtained in § 49 calculate the combining weight of zinc referred to hydrogen as unity. Repeat the experiment, substituting for the sulfuric acid a solution of hydrochloric acid, prepared by adding 1 volume of the laboratory acid (density 1.12) to 1 volume of water. Compare the results.

**118. The combining weight of magnesium.** Determine the combining weight of magnesium, referred to hydrogen as unity, by dissolving the metal in dilute hydrochloric acid and measuring the hydrogen evolved. About 0.5 g. of magnesium ribbon, prepared as directed in § 44, should be used. The hydrochloric acid is prepared by adding 1 volume of the laboratory acid (density 1.12) to 4 volumes of water. The apparatus used is shown in Fig. 27.

**119. The combining weight of aluminium.** Follow the same method as was used in the determination of the combining weights of zinc and magnesium. The hydrochloric acid used is prepared by diluting 2 volumes of the laboratory acid with 1 volume of water.

**120. Determination of molecular weights.** *Molecular weight of chloroform.* (The following experiment will be performed by the laboratory instructor. The students will answer all questions and make the calculations. Before performing the experiment, read over the directions and discuss the method until you thoroughly understand it.) Obtain from the store-room the Victor Meyer apparatus shown in Fig. 49. This apparatus consists of two glass tubes, the smaller one of

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which is suspended in the larger, as represented in the diagram. The liquid in the outer tube *D* is water. In the bottom of the inner tube *A* is placed a little asbestos fiber or sand. The graduated tube *E* has a capacity of about 50 cc. Arrange the apparatus as shown in the figure, except that the tube *E* is not brought over the exit tube *C*. Tightly stopper the tube *A* with a rubber stopper provided with a stopcock *B*, and heat the water in *D* to boiling; then regulate the flame so that a uniform heat is applied, the heat being sufficient to keep the water boiling. The air in tube *A* is heated by the steam, and some of it escapes through *C*, bubbling up through the water. While the heat is being applied, wrap the end of a fine platinum wire 5 or 6 cm. in length about the neck of the little glass-stoppered bottle *F*, suspend the bottle from the beam of the balance, and weigh it. Completely fill the bottle with chloroform, insert the stopper, carefully wipe the bottle to remove any of the liquid adhering to the outside, and reweigh. Now observe whether the air is still escaping from tube *C*. If not, open the stopcock *B*, remove the stopper and stopcock *B*, and, after loosening the stopper in the little glass bottle, hold the bottle in a vertical position with the platinum wire and drop it into the tube *A*. As quickly as possible insert the stopper and close *B*. Before any gas can escape, bring the tube *E* (which must be completely filled with water) over the end of the tube *C* and clamp it in this position. The bottle *F* drops to the bottom of the tube, and the chloroform, on account of its low boiling point ( $61^{\circ}$ ), is vaporized by the heat. The vapor formed expels a definite volume of air, which is caught in the graduated tube *E*. The heating is continued until the gas ceases to escape.

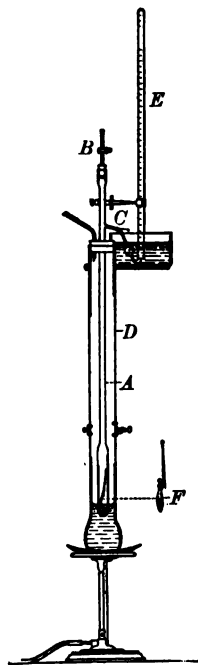


FIG. 49



The stopcock *B* is then opened and the heat withdrawn. Insert the values in the following table:

Weight of chloroform taken . . . . .
Volume of air collected in <i>E</i> . . . . .
Temperature of air in <i>E</i> . . . . .
Pressure to which the air in <i>E</i> is subjected . . .

It is evident that the volume of the air in *E* must be equal to the volume occupied by the vapor formed by the chloroform, provided that the vapor is measured under the conditions of temperature and pressure to which the air in the tube is subjected. Knowing, therefore, the weight of the chloroform taken and the volume of vapor formed under definite conditions, it is easy to calculate the weight of 22.4 liters of the vapor under standard conditions.

By analysis it is possible to show that chloroform has the following composition: carbon, 10.04 per cent; hydrogen, 0.84 per cent; chlorine, 89.12 per cent. From your results determine the formula of chloroform. Why is it necessary to know the molecular weight in order to determine the formula?

*Notes and queries.* The stopper of the little bottle *F* containing the chloroform is loosened before dropping the bottle into the tube, in order that the vapor of the chloroform may easily escape. In performing the experiment the heat must be applied uniformly until the experiment is completed (why?); hence the flame should be protected from drafts of air. The amount of chloroform introduced must be small (about 0.13 g.); otherwise the volume of the vapor will be so large that the tube *E* will not hold the displaced air. Could other liquids be substituted for water in the outer tube? Before performing the experiment the tube *A* must be perfectly dry (why?). To remove any vapor, insert a long, narrow glass tube and force a current of dry air through the tube, at the same time applying a gentle heat. Before measuring the volume of the air in *E*, bring the water within the tube to the level of the water outside, if necessary transferring the tube to a deep cylinder to accomplish this.



## CHAPTER XXI

### SOME HYDROCARBONS

**121. Methane.** *a.* Weigh out approximately 10 g. of fused sodium acetate and double that weight of soda lime (a mixture of calcium oxide and sodium hydroxide). Grind the two together in a mortar and put the mixture in a hard-glass test tube furnished with a stopper and a delivery tube (the oxygen generator of § 15). Support the tube with a clamp and heat gently, gradually increasing the heat. After the air has been expelled from the apparatus, collect two or three bottles of the gas over water.

*b.* Note the color, odor, and solubility of the gas. Hold a lighted match to the bubbles as they escape from the water. Pour a little bromine water into one of the bottles, place a stopper in the bottle, and shake vigorously. Is the color of the bromine changed? Set fire to the gas in the second bottle and, after it has burned, test the residual gas for the presence of carbon dioxide. Half fill a bottle with methane and then displace the remaining water with air. Quickly bring the mouth of the bottle over a flame. What connection is there between this experiment and a mine explosion?

**122. Ethylene.** *a.* Arrange a 250-cc. generating-flask on a sand bath and connect it with a gas-washing bottle containing dilute sodium hydroxide and provided with a delivery tube for collecting over water. Pour 10 cc. of alcohol very slowly into 60 cc. of concentrated sulfuric acid, stirring constantly during the mixing; pour the mixture into the generating-flask and insert the stopper. Carefully heat the mixture until there is a steady evolution of gas. If the heating is too rapid or the temperature reached is too high, the mixture may foam





badly and the alcohol be carbonized. Remembering that some free carbon is usually formed in the reaction, what gases other than ethylene would you expect to be formed? How would these act with sodium hydroxide in the wash bottle?

*b.* Collect two bottles of the gas and test its combustibility and its conduct towards bromine water. How does it compare with methane?

**123. Acetylene.** *a.* With your pliers pick up a small piece of calcium carbide and quickly insert it under the mouth of a test tube filled with water and inverted in a beaker of water. What action takes place? Test the reaction of the water with litmus paper. Holding the test tube in an inverted position (why?), remove it from the water and quickly ignite the gas at the mouth of the tube. How does the flame compare with that of burning ethylene and burning methane?

*b.* Collect another test tube full of the gas, quickly pour into it about 1 cc. of bromine water, close the mouth of the tube with the thumb, and shake it vigorously. Is the bromine absorbed by the acetylene?

**124. Iodoform.** Pour 2 or 3 cc. of a solution of sodium hydroxide into a test tube, add a crystal or two of iodine, and then a few drops of alcohol. Warm slightly and observe the changes which occur. What color does the solution become? Is there a precipitate? What do you notice in regard to the odor? The product is iodoform ( $\text{CHI}_3$ ), and its formation may be used as a good test for alcohol (though some other substances, when treated with iodine and an alkali, also give iodoform).



## CHAPTER XXII

### FUEL GASES; FLAMES; MEASUREMENT OF HEAT

**125. Nature of a flame.** Prepare some charcoal by heating pieces of splints 3 or 4 cm. in length in the bottom of a test tube. Note and account for the difference between the combustion of the splint and that of the charcoal. What are the conditions necessary for the production of a flame? Light a candle and place it so that the flame is against a black background and is not disturbed by air drafts; then note the different cones in the flame. Test the relative temperatures of different parts of the flame by means of narrow strips of splints. Draw a diagram showing the different parts of the flame. Extinguish the candle flame and hold a lighted splint 2 or 3 cm. from the wick in the little column of smoke. The candle is relighted. What does the experiment prove?

**126. Illuminating gas.** Repeat the experiment of § 50, *a*, substituting some pulverized soft coal for the bits of splint. Test the inflammability of the gas. Is its flame luminous?

**127. Products of combustion.** What elements constitute the main parts of ordinary fuels? What products form when these elements burn in air or oxygen? Devise simple experiments to show the presence of these products in the gases evolved by the burning candle. Account for the moisture deposited on a lamp chimney when the lamp is first lighted.

**128. Kindling temperature.** What is meant by the kindling temperature of gases? Press a piece of wire gauze halfway down on a Bunsen flame. Notice that the flame does not extend above the gauze. Is this due to the absence there of combustible gases? Test for their presence by means of a lighted splint. Account for the results. Turn off the gas,



then turn it on and ignite it over a piece of wire gauze held horizontally 4 or 5 cm. above the top of the burner. Note the results and explain. How does the miner's safety lamp prevent explosions? Hold a porcelain dish in a small, luminous Bunsen flame. Account for the deposition of carbon (carbon black). Does the nonluminous flame deposit carbon? To what is the luminosity of the flame due?

**129. Bunsen flame.** Hold a wire horizontally in the base of the Bunsen flame for two or three seconds and note the results. In the same way determine the relative temperatures of various parts of the flame. Turn the gas down until the flame is 7 or 8 cm. in height, then quickly thrust a piece of white cardboard, about 10 cm. in height, *vertically* through the center of the flame, the lower edge of the cardboard resting against the top of the burner. Remove the cardboard before it is ignited, and note from the scorched portions the relative temperatures of different parts of the flame. Draw a diagram to illustrate your results.

That the center of the base of the flame contains the unburned gas may be shown by holding in it the end of an inclined glass tube 12 or 15 cm. in length

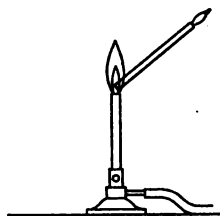


FIG. 50

(Fig. 50) and igniting the gas at the upper end of the tube.

**130. Oxidizing-flame and reducing-flame.** Ask the instructor to show you how to produce each of these flames by means of the blowpipe. Then heat in the reducing-flame a small amount of an intimate mixture of sodium carbonate and lead oxide ( $\text{PbO}$ ) placed in a small cavity in a piece of charcoal. Have you any evidence of the reduction of the oxide? Try the effect of the oxidizing-flame on a small piece of metallic lead on charcoal. Describe the results. Carefully distinguish between an oxidizing-flame and a reducing-flame. To what does each owe its peculiar property?

**131. Heat of neutralization.** Prepare a calorimeter as represented in Fig. 51. The calorimeter beaker *A* should hold approximately 400 cc., should be of light weight, and, if



possible, should be without a lip. The larger beaker, *B*, should hold at least 1 liter. Provide three flat corks of equal thickness to serve as supports for *A*; a stirrer *C* made from a piece of light-weight rod and bent into a ring at the end; and a thermometer *D* graduated to one tenth of a degree. Cut a cardboard cover *E*, circular in form, to fit over *A* and inside *B*; also a cover *F* for *B*, which may be left square. Punch a hole in the middle of each cover, through which the thermometer may be pushed, but small enough to hold the latter in place. A second hole must also be punched in each cover to provide for the handle of the stirrer.

In a graduated cylinder, measure as accurately as possible 150 cc. of a normal solution of NaOH, and pour it into *A*; measure also 150 cc. of a normal solution of HCl, and pour it into a second beaker. Read the temperature of the two solutions at frequent intervals until they have come to exactly the same constant point, warming the cooler one, if necessary, with the hand.

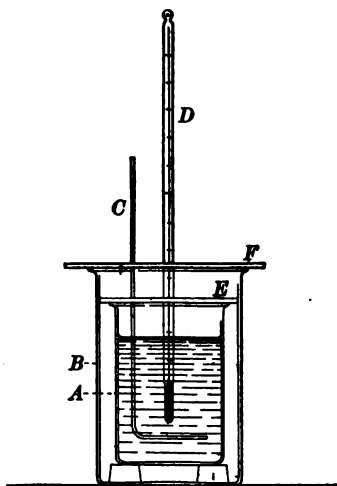


FIG. 51

Make a note of this temperature, reading it as closely as possible. Arrange the thermometer and the stirrer through the holes in the covers so that all may be put in place together; then pour the acid into the alkali and at once adjust the covers and the thermometer. Maintain a slow but steady motion with the stirrer, watching the rise in temperature very closely. Make a note of the highest temperature reached.

The specific heat of the dilute solution may with little error be taken as equal to that of water (unity), so that the rise in temperature, multiplied by the volume of the solution (in this case 300 cc.), will give the *apparent* heat of the





reaction. Some heat has been absorbed by the calorimeter beaker, the thermometer, and the stirrer, and this must be determined and added to the apparent heat. If the materials are of the character specified, experiment has shown that the heat absorbed will be about 12 cal., and this value may be assumed with little error. The constant may be calculated by determining the weight of the portion of the beaker and stirrer in contact with the water, multiplying this by 0.19 (the specific heat of glass), and to this adding the *volume* of the submerged part of the thermometer, multiplied by 0.49.

Having in this way found the heat of neutralization of 150 cc. of normal NaOH, calculate the heat of neutralization of 1 gram-molecular weight of the base (1000 cc.). The value determined by accurate experiment is 13,700 cal.

**132. Heat of solution.** Accurately measure 300 cc. of pure water into the calorimeter beaker *A* (Fig. 51). Grind about 15 g. of potassium nitrate to a very fine powder in a mortar, and place it in a test tube. Weigh the tube and nitrate very accurately and immerse the lower end of the tube in the water in the calorimeter until the nitrate has come to the temperature of the water (about fifteen minutes). Make a note of the temperature. Remove the tube with as little loss of water as possible, roughly dry it with a towel, and at once pour most of the nitrate into the water. Quickly replace the covers, as in § 131, stir vigorously, and note the lowest temperature recorded. Then weigh the tube and the remaining nitrate, deducting this weight from the original one to get the weight of the nitrate added. From the data so secured, together with the calorimeter constant, calculate the heat of solution of 1 gram-molecular weight of potassium nitrate. Berthelot found this to be -8300 cal.



## CHAPTER XXIII

### CARBOHYDRATES; ALCOHOLS; SOAPS

**133. Preparation of Fehling's solution.** The most common test for sugars is their reaction with the so-called "Fehling's solution." This solution is prepared as follows: Dissolve 3.5 g. of copper sulfate crystals in water and dilute to 50 cc. Pour the solution into a bottle and label it "Solution A." Dissolve 5 g. of sodium hydroxide and 17.5 g. of sodium-potassium tartrate (Rochelle salts) in about 40 cc. of water and dilute to 50 cc. Pour this into a bottle and label it "Solution B."

**134. Action of Fehling's solution on dextrose.** Pour into a test tube about 3 cc. of each of the above solutions marked "A" and "B." When thoroughly mixed, the resulting solution should be deep blue but perfectly clear. Heat nearly to boiling, add a few drops of a solution of commercial glucose, and continue the heating. The copper compound in the solution is reduced to cuprous oxide by the dextrose present, and this separates in the form of a red or yellow solid. Levulose will act in the same way. Test samples of candy, honey, and molasses for the presence of these sugars.

**135. Action of Fehling's solution on cane sugar.** In a similar way try the action of cane sugar on the solution. Note that the pure sugar does not reduce the alkaline copper compound. Now dissolve about 1 g. of the sugar in 10 cc. of water. Add 4 or 5 drops of concentrated hydrochloric acid and slowly heat to boiling. Set aside for about five minutes, then cool and neutralize the acid in the solution by adding a concentrated solution of sodium carbonate until the resulting mixture is just alkaline to litmus paper. Test this with Fehling's solution according to above directions. Account for the result.



**136. The test for starch.** The presence of starch is best shown through its action on iodine (§ 104).

**137. Alcohol.** The formation of alcohol and carbon dioxide from glucose may be shown as follows: About 100 g. of glucose is dissolved in a liter of water in flask *A* (Fig. 52). This flask is connected with the bottle *B*, which is partly filled with limewater. The tube *C* contains solid sodium hydroxide. A little baker's yeast is now added to the solution in flask *A*, and the apparatus is connected as shown in the figure. If the temperature is maintained at about 30°, the reaction soon begins, but it will not be completed for several days. The experiment can be finished at a later laboratory period. The bubbles of gas escape through the limewater in *B*. A precipitate of calcium carbonate soon forms in the limewater, showing the presence of carbon dioxide. The sodium hydroxide in tube *C* prevents the carbon dioxide in the air from acting on the limewater. The alcohol remains in the flask *A*. Connect the flask with a condenser (Fig. 24) and distill about 10 cc. of the solution. Pour this into an evaporating-dish and see whether it will burn. Test a few drops with the iodoform test (§ 124).

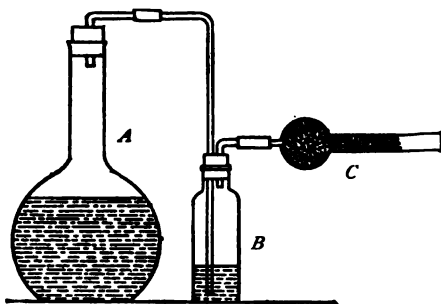


FIG. 52

**138. Acetic acid.** *a.* Place about 10 g. of sodium acetate in a small flask (about 250 cc.) provided with a stopper and delivery tube. Pour 5 cc. of concentrated sulfuric acid into an equal volume of water, and add the solution to the flask. Arrange the apparatus so that the delivery tube just touches the surface of a little water contained in a beaker, and then gently heat the mixture in the flask until it has boiled for a short time and several drops of distillate have passed over into the water in the beaker.



**b.** Notice the odor of the distillate. Test its reaction with litmus. Add sodium hydroxide to the distillate until it is just neutral, and then pour in a few drops of a solution of ferric chloride ( $\text{FeCl}_3$ ). The solution becomes blood red, and this reaction constitutes a test for acetic acid.

**139. Esters.** Place 1 or 2 g. of sodium acetate in a test tube. Add 1 or 2 cc. of alcohol and about 1 cc. of concentrated sulfuric acid, and warm gently. The product of the reaction is ethyl acetate ( $\text{CH}_3\text{CO}_2\text{C}_2\text{H}_5$ ). Notice the pleasant odor. The formation of this substance with its characteristic odor may be used as a test for an acetate or for acetic acid.

**140. Determination of the acidity of vinegar.** Introduce 5 cc. of vinegar into a small beaker and dilute with about 50 cc. of water. Add 2 drops of an alcoholic solution of phenolphthalein and then slowly run in a solution of sodium hydroxide until the solution is neutral, as in § 71.

If a solution of sodium hydroxide of known strength is used, it is evident that one may in this way determine the percentage of acetic acid in the vinegar. The laws of most of the states require a minimum of 4 per cent of acetic acid in all vinegars.

**141. Saponification and the preparation of soap.** Add 10 cc. of alcohol to 4 or 5 g. of cottonseed oil in an evaporating-dish. To the resulting mixture add about 1 cc. of a 50 per cent solution of sodium hydroxide in water. Evaporate carefully, stirring the mixture constantly until the odor of alcohol can no longer be detected. Write the equation for the reaction on the supposition that the oil is composed of palmitin. What remains in the dish? Add 75 cc. of cold water, stir well, and filter. Add a little of the resulting filtrate to test tubes containing solutions of calcium sulfate, magnesium sulfate, and sodium sulfate respectively. Explain the results. Account for the fact that soaps do not lather freely in hard waters.





## CHAPTER XXIV

### THE PHOSPHORUS FAMILY

**142. Phosphorous acid.** Pour into a test tube about 0.5 cc. of phosphorus trichloride (hood) and add a little water, a drop at a time. What gas is evolved (R)? Finally, add about 5 cc. of water, pour the liquid into an evaporating-dish, and evaporate to a sirupy mass. Dilute this with a little water, transfer the solution to a test tube, and add a few drops of a solution of silver nitrate. Boil the resulting mixture. A black precipitate forms. Explain.

**143. Phosphoric acid.** *a.* Add, in very small portions, about 1 g. of phosphorus pentoxide to 15 cc. of cold water. To 1 cc. of the solution add a solution of silver nitrate until a precipitate forms. The precipitate is  $\text{Ag}_3\text{PO}_4$ . Explain. Pour the remainder of the original solution into an evaporating-dish and evaporate to a sirupy mass. Add about 10 cc. of water and carefully neutralize the resulting solution with sodium carbonate.  $\text{Na}_2\text{HPO}_4$  is formed. Test the solution with silver nitrate. The equation for the reaction is as follows:



*b.* Test a solution of normal sodium phosphate with litmus paper. Explain. Add a little silver nitrate (R).

*c.* Add a few drops of magnesia mixture to a solution of disodium phosphate. (Magnesia mixture is prepared by dissolving 1 g. of magnesium chloride crystals and 3 g. of ammonium chloride in 10 cc. of water and adding 1 cc. of ammonium hydroxide solution.) The precipitate has the composition  $\text{MgNH}_4\text{PO}_4$ . Advantage is taken of this reaction in determining the amount of magnesium in various substances.



*d.* With litmus paper test the reaction of disodium phosphate and of silver nitrate. Now add a solution of silver nitrate to one of disodium phosphate until an appreciable precipitate has been formed, and test the mother liquor. How do you account for the reaction?

*e.* Add a few drops of a solution of disodium phosphate to 5 cc. of a solution of ammonium molybdate (side shelf). A bright yellow precipitate of complex formula slowly forms (§ 88, *b*).

**144. Pyrophosphates.** Apply a gentle heat to 1 or 2 g. of disodium phosphate placed in a porcelain crucible. Gradually increase the heat to the full extent and continue the heating for about ten minutes. When the crucible has cooled, dissolve the product in cold water and test the solution with silver nitrate solution. Compare with the product obtained by adding silver nitrate to disodium phosphate which has not been heated. Explain.

**145. Metaphosphates.** *a.* Repeat the experiment of § 144, using microcosmic salt ( $\text{NaNH}_4\text{HPO}_4$ ) instead of disodium phosphate. (This salt, on being heated, acts just as does  $\text{NaH}_2\text{PO}_4$ .) Explain.

*b.* Compare the results obtained by adding silver nitrate to solutions of the salts of each of the three phosphoric acids.

*c.* Make a loop on the end of your platinum wire and in this form a bead like the borax bead (§ 151, *a*), using microcosmic salt instead of borax (*R*). Of what will the clear bead consist? What color is given to the bead by rubbing it in a little copper oxide and reheating (*R*)? Test the color occasioned by a compound of cobalt and one of chromium. What is formed when sodium metaphosphate dissolves these oxides? Will sodium nitrate form similar compounds? Why? Could sodium metaphosphate be used as a flux in brazing?

**146. Arsenic.** *a.* Note the physical properties of arsenic. Heat (hood) a bit of it half as large as a grain of wheat, on charcoal, with a blowpipe. Explain.

*b.* Seal the end of a piece of glass tubing about 10 cm. in length and 6 or 7 mm. in diameter. Introduce into it an

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amount of arsenious oxide equal in bulk to a grain of wheat. Cover this to a depth of 2 or 3 cm. with somewhat finely powdered charcoal which has been strongly heated in a porcelain crucible. See that the inner surface of the tube above the charcoal is perfectly clean. Incline the tube and heat the *upper* portion of the charcoal to a high temperature; then, while maintaining the charcoal at this temperature, gradually bring the lower part of the tube also into the flame (Fig. 53). The upper part of the tube must be kept as cool as possible. The arsenious oxide is changed into a vapor, which passes over the hot charcoal. Account for the result (R). Cut the tube as near the bottom as possible and remove the charcoal; then, inclining the tube, apply a very *gentle* heat to the portion of it which contains the dark mirror. Note that small white crystals are slowly deposited in the colder portions of the tube (R). Note their form.

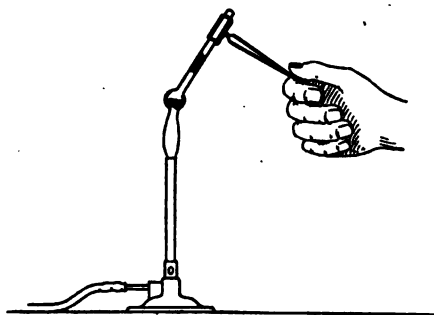


FIG. 53

*c. Marsh's test (hood).* The arsine formed in this experiment is very poisonous, and great care must be taken to prevent its escape into the air of the laboratory.

Arrange an apparatus according to Fig. 54, consisting of the generator *A*, a calcium chloride drying-tube *B*, and a clean hard-glass tube *C* about 30 cm. long and 8 mm. in diameter, drawn out to a jet at the end. (Use the blast lamp in making the jet.) Generate hydrogen by the usual method and, after taking the general precautions (§ 24, *d*), ignite it as it escapes from the glass jet *D*. Sufficient acid is added from time to time to cause a gentle evolution of the gas. Now apply a strong heat to the hard-glass tube at a place near its middle, using the wing-top burner. After a few minutes note whether any deposit forms just beyond the heated portion of the tube.



If none forms, the materials are free from arsenic. Now ask the instructor to add to the generator 2 drops of a dilute solution of arsenious oxide in hydrochloric acid, rinsing it down the funnel tube with a little water. Continue the heating of the hard-glass tube at the same place. Note the deposit formed on the sides of the tube. Withdraw the heat and hold the lid of a porcelain crucible in the flame. A black deposit of arsenic forms. Test the solubility of this in a solution of sodium hypochlorite. Note the results. Cut the tube containing the deposit so as just to remove the jet, and, inclining it,

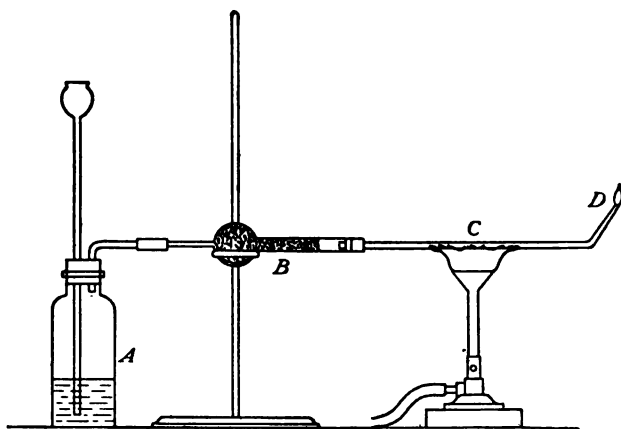


FIG. 54

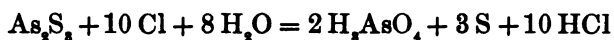
apply a *gentle* heat, as in *b*. Account for the results and write the equations for all the reactions involved in the experiment.

**147. Arsenious oxide.** Place a very little arsenious oxide in a test tube and add a few cubic centimeters of a solution of sodium hydroxide, heating the solution gently. Does the oxide dissolve (R)? What do you infer as to the character of arsenious oxide? Save the solution for § 148. Repeat the experiment, substituting hydrochloric acid for the sodium hydroxide (R). What would this experiment suggest as to the nature of arsenious oxide? Set the test tube aside and note the crystals which separate. They are  $\text{As}_2\text{O}_5$ . What light does this throw on the nature of arsenious oxide?





**148. Arsenious sulfide.** Pass a current of hydrogen sulfide through the two solutions just described. What difference in conduct do you note? What does the yellow color indicate? Is arsenious sulfide soluble in water? Why does it not precipitate in the first solution? Now add hydrochloric acid to this solution until it is acid in reaction. What change takes place? How do you explain it? What is the precipitate? Collect it by filtering through a small filter, and transfer the precipitate to a test tube. Cover it with concentrated hydrochloric acid, add a bit of potassium chlorate, and heat to boiling (hood). The sulfide is converted into the soluble arsenic acid, in accordance with the following equation:



What is the source of the chlorine? What is the function of the potassium chlorate? Now heat the solution containing the arsenic acid until the chlorine is all expelled, add ammonium hydroxide until the solution is alkaline, and then enough more to increase the volume by one third. Filter unless the liquid is clear. Add a few drops of magnesia mixture and shake the solution vigorously. The precipitate is  $\text{MgNH}_4\text{AsO}_4$ . Compare with the action of magnesia mixture on phosphoric acid (§ 143, c).

Tabulate the names and the formulas for the acids of arsenic. How do they compare in composition with those of phosphorus?

**149. Antimony.** *a.* Repeat § 146, *a*, substituting antimony for the arsenic. Note the results.

*b.* Repeat § 146, *c*, substituting 2 or 3 drops of a solution of a compound of antimony for the arsenious oxide. Compare the results of the two experiments (R).

*c.* Introduce a bit of antimony no larger than a grain of wheat into a test tube, and add about 2 cc. of hydrochloric acid. Does the antimony dissolve? Why? Add 2 or 3 drops of nitric acid (hood). After the antimony has been dissolved, pour the solution into a beaker containing about 100 cc. of water (R). Half fill a test tube with the resulting



mixture, add hydrochloric acid, a drop at a time, until the solution just clears (R). Now pass hydrogen sulfide through the clear solution until an orange-colored solid forms (R).

*d.* Boil a small amount of powdered antimony with concentrated nitric acid (hood) until it is changed into a white powder. Dilute the mixture with water, filter, and wash the residue on the filter paper with water. Convince yourself that the residue is not a nitrate. It is a hydrated oxide of variable formula, but may be considered to have the formula  $\text{Sb}_2\text{O}_3$ . Dissolve a portion of it in sodium hydroxide (R). Dissolve another portion in hydrochloric acid (R).

**150. Bismuth.** *a.* Repeat § 146, *a*, substituting bismuth for arsenic. Contrast the action of heat on arsenic, on antimony, and on bismuth.

*b.* Repeat § 149, *c*, substituting bismuth for antimony and using nitric acid alone as the solvent.



## CHAPTER XXV

### BORON AND SILICON

151. **Borax bead.** *a.* Make a little loop on the end of a platinum wire and heat it to redness in a Bunsen flame ; then quickly bring the loop in contact with some borax and *reheat*. The borax adhering to the loop will swell up (why ?) and finally form a clear glass bead. Now dip the bead into a solution of a cobalt compound and reheat thoroughly (R).

*b.* Repeat the experiment, substituting a compound of chromium for the cobalt solution.

152. **Boric acid.** *a.* Dissolve 5 g. of borax in 15 cc. of *boil-*ing water. Test the solution with litmus paper. Explain. Add to the hot solution 5 cc. of concentrated hydrochloric acid. Cool the solution, filter off the precipitate (R). Compare the precipitate with borax as to solubility in alcohol.

*b.* Place 1 cc. of a solution of borax in an evaporating-dish and add a few drops of sulfuric acid (R) and 2 or 3 cc. of alcohol. Set fire to the alcohol and watch for a green color in the flame. This is a test for boric acid. Will borax act in the same way if no sulfuric acid is added ?

*c.* Heat a little boric acid in an iron crucible until a clear liquid is formed (R).

153. **Borax.** Prepare a *concentrated* solution of borax and add a few drops of a solution of silver nitrate. The precipitate is silver borate (R). Add a *few drops* of the concentrated solution of borax to a test tube half full of water, and then test with silver nitrate. The precipitate is silver oxide (R). Compare it with the precipitate formed by the action of very dilute sodium hydroxide on silver nitrate (R). How do you account for the different actions of borax on silver nitrate ?



**154. Silica.** *a.* Mix about half a gram of fine sand with 3 or 4 times its weight of solid sodium hydroxide. Place the mixture in an iron dish, and heat until fusion has taken place and the fused mass has again become solid (R). Then dissolve the product in hot water.

*b.* Pour half of the solution into a test tube and add an excess of hydrochloric acid. Allow it to become perfectly cold. What is the jellylike substance?

*c.* Transfer the remainder of the solution to an evaporating-dish, acidify with hydrochloric acid (R), evaporate to dryness, and heat the dish gently with the bare flame (R). Is the product soluble in water? in acids? in alkalis? in fused sodium hydroxide?





## CHAPTER XXVI

### COLLOIDS

**155. Preparation of colloids.** *a.* Prepare 4 or 5 cc. of a saturated solution of sulfur in alcohol and pour it into 100 cc. of water. The sulfur forms a fine white dispersion that will not settle. Filter the mixture (?).

*b.* To 10 cc. of water in a test tube add 3 drops of a dilute solution of gold chloride (6 g. crystallized  $\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$  per liter). Heat to boiling and add 1 or 2 cc. of a dilute solution of formalin as a reducing agent (3 cc. commercial formalin per liter of water). The solution assumes a purplish color due to metallic gold. Compare the appearance viewed by transmitted light with that by reflected light.

*c.* To 100 cc. of water add 2 or 3 drops of the solution of gold chloride. After thorough shaking, add a few drops of a dilute solution of tannin (about 0.1 g. per liter) and heat to near boiling. If the color is too faint repeat the addition of the two reagents. How does the color compare with that obtained in *b*? How do you account for the difference? The tannin acts both as a reducing agent and a protecting colloid. To the red solution add 1 cc. of a concentrated solution of sodium chloride. What change do you notice?

**156. Preparation of colloids by double decomposition.** *a.* Prepare a little antimony sulfide by dissolving a small grain of antimony oxide or antimony chloride in 5 cc. of dilute hydrochloric acid and adding a solution of hydrogen sulfide. The orange-colored precipitate has the composition  $\text{Sb}_2\text{S}_3$ . When the precipitate has settled, is the liquid colored? Set the tube containing it aside for reference.



**b.** Dissolve a little tartar emetic (which contains antimony as a salt of a *weak* acid), not exceeding a wheat grain in bulk, in about 100 cc. of water, and add a few drops of a solution of hydrogen sulfide, taking care to avoid an excess. (The resulting solution should not have the odor of hydrogen sulfide.) What change do you notice? Filter a little of the solution. Is the filtrate colorless? Is a precipitate left on the filter? Boil 2 or 3 cc. of the filtrate and again filter. Does boiling cause the antimony sulfide to separate? Reserve the remainder of the solution for §§ 158, 159, 160.

**157. Preparation of colloids by hydrolysis.** **a.** To 100 cc. of water in a beaker add enough of a solution of ferric chloride to give a decidedly yellow color. Slowly heat the solution to boiling and note the change in color. The salt is hydrolyzed with the formation of colloidal ferric oxide. View the color by reflected and then by transmitted light. Compare the latter with the color of ferric oxide precipitated from dilute ferric chloride by ammonium hydroxide. Save the solution for §§ 157, 158, 159.

**b.** Dissolve 2 or 3 g. of sodium acetate in 50 cc. of water. Now add 1 cc. of a solution of ferric chloride. What new salts should be formed? Should either of these undergo extensive hydrolysis? Do you obtain a precipitate? What is the color of the solution? This is largely due to the presence of colloidal ferric oxide. Save this solution for §§ 158, 159, 160.

**158. Coagulation of colloids.** Heat to boiling 5 cc. of the colloidal solutions obtained in 156, **b**, and 157, **a** and **b**. Is the colloid precipitated? To 5 cc. of each add about half a gram of ammonium chloride or calcium chloride and shake the solution vigorously. Do any of the solutions give a precipitate? To 5 cc. of each add 1 drop of concentrated sulfuric acid. Would you expect this reagent to precipitate ferric oxide? Explain. Remembering that fermentation often produces acids, how can you explain the curdling of milk on souring?

**159. Protective colloids.** Dissolve a few small pieces of agar (a colloidal material resembling gelatin) in about 10 cc. of



hot water. Add equal portions of this solution to portions of the colloidal solutions of antimony sulfide and of ferric oxide. With these solutions repeat the experiments of § 158. Does the presence of the agar interfere with coagulation? It is often the case that the presence of organic colloids interferes with precipitation tests in chemical analysis. Such colloids are then called protective colloids.

**160. Precipitation of one colloid by another.** Sometimes one colloid will act upon another, causing mutual precipitation. Treat some of the colloidal solution of iron oxide with an equal volume of the antimony sulfide, taking care that no free hydrogen sulfide is present. Does a precipitate form? Filter some of the solution. Is the filtrate red like the iron, or orange like the antimony?

**161. Adsorption.** *a.* Fold a small filter paper to fit a funnel and place upon it a layer of bone black 2 or 3 cm. thick. Wet it thoroughly with water and let it drain. Prepare 50 cc. of a solution decidedly colored by blue litmus, congo red, or some other dye. Slowly filter the colored solution through the bone black, repeating several times. Is the color removed?

*b.* Repeat the experiment, using a dilute solution of ammonia instead of the dye. Can you detect ammonia in the filtrate? How does it affect red litmus?

**162. Emulsions.** *a.* In a 250-cc. flask pour 5 cc. of a 5 per cent solution of hard soap in water. Add benzene in successive portions as follows: 5, 10, 20, 20, 20, 20, 30, 50 cc., shaking the stoppered flask vigorously after each addition. A stiff emulsion should be obtained consisting of about 97 per cent benzene emulsified in 3 per cent of water by a very little soap.

*b.* Shake together 20 cc. of raw linseed oil and 20 cc. of water, adding the water in small portions at a time. The oil always contains enough rosin to act as the emulsifying agent.

**163. Gels.** *a.* Break up some dry gelatin or agar into very small pieces and place them in a test tube to a depth of about 2 cm. Add 10 cc. of water and heat nearly to boiling until a clear solution is obtained. Immerse the test tubes in ice water



for a time. What change do you note? Now put the tube in hot water. Does the gel liquefy? Once more cool. Does the gel form again? What is such a gel called?

*b.* To 10 cc. of water glass of density 1.1 add 10 drops of concentrated sulfuric acid. Shake the solution thoroughly and set aside if necessary until the next period. Tap the test tube gently and note the vibrations of the firm gel of silicic acid.

*c.* Immerse the tube containing the gel in hot water. Does the gel liquefy? With a glass rod dig out a little of the gel and see if you can dissolve it in water. What is such a gel called?





## CHAPTER XXVII

### GENERAL METHODS FOR PREPARATION OF COMPOUNDS

**164. General methods.** *a. Chlorides.* How can you prepare zinc chloride from zinc? from zinc carbonate? from zinc oxide (R)? How can you prepare calcium chloride from calcium carbonate? What chlorides are insoluble? Prepare small amounts of each (R) and note their physical properties.

*b. Sulfides.* Describe the different ways in which sulfides have been prepared in the laboratory. Devise a method for preparing the following insoluble sulfides: lead sulfide, silver sulfide, antimony sulfide, zinc sulfide, manganese sulfide. Prepare (hood) small amounts of each in test tubes (R).

*c. Nitrates.* How can you prepare copper nitrate from copper? from copper carbonate? from copper hydroxide (R)? Are any of the nitrates insoluble? What is the effect of heat on copper nitrate? on lead nitrate? on ammonium nitrate?

*d. Nitrites.* Recall the preparation of sodium nitrite (R).

*e. Sulfates.* The following sulfates have been prepared in previous exercises: zinc sulfate, sodium sulfate, copper sulfate, iron sulfate. Write the equations for the reactions involved in the preparation of each. What sulfates are insoluble? Prepare small amounts of each, noting the color and writing the equations for the reactions involved in each case.

*f. Sulfites.* Recall the method used in the preparation of sodium sulfite. How can you distinguish between sulfates and sulfites?

*g. Carbonates.* What carbonates are soluble? Give a general method for the preparation of the soluble carbonates; give a general method for the preparation of the insoluble carbonates. Prepare calcium carbonate by two different



methods (R). How could you prepare from calcium carbonate the following compounds: calcium chloride, calcium sulfate, calcium nitrate, calcium oxide (R)? Why are carbonates so readily decomposed by acids?

*h. Phosphates.* Recall the formulas and the names of the three phosphoric acids, also the action of silver nitrate on their salts.

*i. Oxides.* Burn a bit of magnesium wire 1 or 2 cm. in length (R). Heat a crystal of lead nitrate in a test tube (R). Heat a small piece of limestone on a wire gauze (R).

*j. Hydroxides.* Hold a piece of lime the size of a walnut in water for a few seconds, then place it on a watch glass and set it aside for a half hour. Note the change (R). Pour into separate test tubes about 1 cc. of a solution of each of the following compounds: ferric chloride ( $\text{FeCl}_3$ ); magnesium sulfate ( $\text{MgSO}_4$ ); copper sulfate ( $\text{CuSO}_4$ ). Add to each solution 2 drops of a solution of sodium hydroxide (R). Now add a few drops of hydrochloric acid to each test tube, and explain the results.

**165. Equilibrium.** *a.* To 3 or 4 cc. of a solution of barium chloride add dilute sulfuric acid, drop by drop, as long as a precipitate is produced. Is the reaction a complete one? In a similar way, to a solution of calcium chloride add a solution of oxalic acid as long as calcium oxalate ( $\text{CaC}_2\text{O}_4$ ) is precipitated. To test the completeness of the reaction, filter off the precipitate and add ammonium hydroxide to the filtrate until it is alkaline in reaction. Does an additional precipitate form? Can it be ammonium oxalate? What is it? Why did it not appear before the ammonium hydroxide was added? If ammonium oxalate is added in excess to calcium chloride, is the reaction complete? Why is it incomplete when the free acid is used?

*b.* Make a list of about a dozen acids which you have studied. What insoluble salts do they form? Prepare an insoluble salt of each (if it forms one) and try its solubility in hydrochloric acid. Which ones should be insoluble? Why? Do your experiments confirm your predictions?



## CHAPTER XXVIII

### THE ALKALI METALS

NOTE. Some preliminary experiments with sodium and sodium hydroxide have been given in Chapter XI. The student should review these carefully in connection with the present chapter.

**166. Action of sodium hydroxide upon soluble salts of other metals.** Try the effect of the solution of sodium hydroxide upon a soluble salt of each of the following: barium, potassium, magnesium, zinc, copper (R). In each case add a few drops at first, and if a precipitate forms, add an excess to see whether the precipitate redissolves. Make a list of the hydroxides soluble in water, and another list of those which redissolve in an excess of sodium hydroxide.

**167. Preparation of sodium chloride.** Dissolve 5 g. of sodium carbonate in 20 cc. of water. Prepare common salt from this (R). How can you be sure that the product contains no unchanged sodium carbonate? Describe the method (R). Treat some of the salt so prepared with sulfuric acid. What gas is evolved (R)?

**168. Purification of common salt.** The impurities most likely to be present are sulfates and chlorides of calcium and of magnesium. Dissolve about 25 g. of common salt in 100 or 150 cc. of water and heat to boiling. Add a solution of barium hydroxide, 1 cc. at a time, as long as you are sure that the precipitate is increasing (R). Without filtering, add a concentrated solution of sodium carbonate, drop by drop, as long as it produces a precipitate. If the solution becomes too turbid for you to be sure of this, filter it. What is precipitated (R)? When the precipitation is complete, filter. What impurity may now be present? Add dilute hydrochloric acid



until the solution is distinctly acid (R), and then evaporate to dryness in an evaporating-dish, using a very small flame toward the end of the process. Test the product for each of the original impurities. Expose some of it to the air for a number of days. Does it become moist?

**169. Sodium thiosulfate.** *a.* Dissolve 12 g. of crystallized sodium sulfite ( $\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ ) in 25 cc. of water and place the solution in a small flask provided with a stopper through which a short piece of glass tubing has been inserted. Add 1.5 g. of finely powdered *roll* sulfur (not flowers of sulfur) and gently boil the mixture until the sulfur has nearly all dissolved. This may require an hour or more. Why do the stopper and the tube largely prevent oxidation by the air? Will the reaction be more rapid if the solution is boiled vigorously rather than very gently? Why? Filter the solution and set it aside to crystallize. If no crystals form by the next day, add a very small crystal of the thiosulfate to start crystallization. Explain. Filter off the crystals and dry them on filter paper. Are they efflorescent or deliquescent?

*b.* To 2 or 3 cc. of the mother liquor add a little hydrochloric acid (R). Prepare a little silver chloride by precipitation and, after washing it with water, add to it 1 or 2 cc. of the thiosulfate solution. Does it dissolve? What use is made of this reaction? Test the solubility of one or two crystals of iodine in water and then add a little thiosulfate solution (R). How is this reaction used in chemical analysis?

**170. Potassium nitrate.** Give the sources of potassium nitrate. Put into a small beaker the theoretical amounts of sodium nitrate and potassium chloride necessary to prepare 10 g. of potassium nitrate. What are these weights? Add 20 cc. of water and boil gently (R). The sodium chloride formed, being little more soluble in hot water than in cold, soon begins to separate. Stir the solution and continue the heating until the volume of the liquid is reduced about one half. Quickly filter the hot solution and set the filtrate aside until cold. Dry the crystals deposited in the filtrate by pressing





them between filter papers. Finally, recrystallize them from as little hot water as possible. Prove the composition of the purified crystals. If the solution is allowed to cool before filtering, will the preparation be a success? Why?

**171. Potassium bromate and potassium bromide.** Dissolve 10 g. of potassium hydroxide in 15 cc. of water in a 100-cc. flask. Weigh out (*in the hood*) the amount of bromine necessary to convert this into a mixture of the bromide and bromate (R). If it is remembered that the density of liquid bromine is almost exactly 3, the liquid may be measured instead of weighed. Add the bromine, a drop at a time, to the hydroxide solution, shaking the flask after the addition of each drop. If the solution becomes very warm, cool it by immersing the flask in cold water. If the hydroxide is impure, there will be an excess of bromine, which will color the solution. In this case add an excess of the hydroxide, a drop at a time, until the solution just becomes colorless. Cork the flask and set it aside until the next laboratory period, then filter off the bromate. Evaporate the filtrate to dryness in an evaporating-dish and heat to a high temperature. What is the residue? Dry the bromate and heat a portion of it in a hard-glass test tube. What gas is evolved (R)?

**172. Potassium chlorate and potassium chloride.** Prepare potassium chlorate and potassium chloride by a method analogous to that of § 171, generating chlorine as in § 94, *e*, and passing it into the solution of potassium hydroxide till the liquid no longer feels soapy. See also § 107, *a*.

**173. Hydrolysis of salts.** Test with litmus paper solutions of the following compounds: sodium carbonate, sodium hydrogen carbonate, potassium carbonate. Account for the results.

**174. Flame tests.** Hold a platinum wire in the Bunsen flame until it ceases to impart any color to the flame; then dip it into a solution of a compound of sodium and hold it in the outer film of the base of a Bunsen flame. Note the color imparted to the flame. Clean the wire by boiling it in dilute hydrochloric acid and repeat the flame test with a compound of potassium.

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**175. Ammonium compounds.** *a.* Recall the action of sodium hydroxide and calcium hydroxide on compounds of ammonium. Place a crystal of some compound of ammonium in a small beaker, and moisten it with 1 or 2 cc. of a solution of sodium hydroxide. Moisten a strip of red litmus paper, stick it on the bottom of a watch glass, and place the latter on the beaker as a cover. Why does the litmus paper turn blue? Will this serve as a test for ammonium salts?

*b.* Pour 2 or 3 cc. of a solution of ferrous sulfate into each of two test tubes. To the one add a few drops of an aqueous solution of hydrogen sulfide; to the other, a few drops of a solution of ammonium sulfide. Account for the results. In preparing sulfides by precipitation, when is ammonium sulfide used in place of hydrogen sulfide? How is it prepared? What changes does it undergo on standing exposed to air?

*c.* Ask the instructor for unknown compounds of sodium, potassium, or ammonium, and see if you can identify them. Recall such reactions of carbonates, sulfates, nitrates, sulfites, sulfides, chlorides, bromides, and iodides as will serve to identify them.



## CHAPTER XXIX

### THE ALKALINE EARTH METALS

**176. Calcium hydroxide.** *a.* Place some small pieces of marble on a piece of wire gauze and apply a strong heat for about fifteen minutes. When cool, drop the residue into 25 cc. of water and stir. Then filter the liquid and divide the filtrate into two parts. Blow exhaled air through one portion (R). Add 1 or 2 drops of ferric chloride to the other portion (R). What other hydroxides would be precipitated by calcium hydroxide?

*b.* Place about 10 cc. of clear limewater in a test tube and conduct carbon dioxide into the solution. What is the precipitate (R)? Continue the process until the precipitate dissolves (R). Divide the solution into two parts. Boil one part for a few minutes (R). What is temporary hardness? Prepare a dilute solution of soap and shake it vigorously in a test tube. Does it form a froth (lather)? Add to it some of the second portion of the solution and again shake. Does it still form a lather? What is the precipitate?

**177. Calcium chloride.** Dissolve 10 g. of marble in hydrochloric acid (R). What does the effervescence indicate? Marble is likely to contain a little carbonate of magnesium and of iron. What would become of these? Add an excess of limewater (R) and filter. Evaporate the solution to a volume of not more than 10 cc. and allow it to crystallize. What is the formula of the crystals? Drain off the mother liquid and evaporate it to dryness. What is the composition of the residue? Expose a small piece of it to the air for an hour and account for the results. Dissolve the crystals in a little water and divide the solution into two portions. To one



portion add a few drops of ammonium carbonate (R); to the other add a few drops of solutions of ammonium hydroxide and disodium phosphate (R).

**178. Calcium sulfate.** *a.* Heat a crystal of gypsum in a test tube (R). Place on a glass plate some metal object, such as a file, which has been smeared with a drop of oil. Pour over the object a thick paste prepared by adding water to plaster of Paris. Set it aside until it hardens, then remove the object and note the result. What causes the paste to harden? For what is plaster of Paris used?

*b.* Shake 1 g. of calcium sulfate with 10 cc. of water in a test tube, filter, and divide the solution into two parts. Test one part for the presence of sulfates. Is calcium sulfate soluble in water? Shake the other part with 1 or 2 cc. of soap solution. Does a froth form? How could such a solution be softened? Distinguish between temporary and permanent hardness of water. To what is each due?

**179. Barium chloride.** Weigh out *accurately* in a porcelain crucible from 1 to 2 g. of small crystals of barium chloride. Place the lid on the crucible and heat the crucible gently for a few minutes, holding the burner in the hand and moving it about so as to apply the heat uniformly. Finally, apply a strong heat for five minutes. When the crucible is cool, weigh. The residue is  $\text{BaCl}_2$ . From your results determine the number of molecules of water of hydration in the crystals.

**180. Analytical reactions.** Place in separate test tubes solutions of a compound of each of the following elements: calcium, barium, strontium. Test the effect of each on the flame by means of a platinum wire, as in § 174. Add a few drops of a solution of potassium dichromate to each. Test the solubility of the precipitates in acetic acid. Add an equal volume of a saturated solution of calcium sulfate to solutions of each, heat to boiling, and set aside until cool. Note all the results. How could you distinguish between compounds of the three elements?





## CHAPTER XXX

### THE MAGNESIUM FAMILY

**181. Magnesium carbonate.** Place 2 or 3 g. of magnesium carbonate (preferably the mineral magnesite) in a hard-glass tube fitted with a delivery tube, and gently heat in the Bunsen flame, passing the gas evolved through clear limewater. What would you judge as to the ease of decomposition of magnesium carbonate in comparison with that of calcium carbonate? Increase the heat, and when the evolution of gas becomes slow, cool the solid product and shake it with water, testing the reaction toward litmus paper.

**182. Magnesium chloride.** *a.* What is the formula of crystallized magnesium chloride? Place a little of the solid in a test tube and heat it gently. Is water given off easily? With blue litmus paper keep testing the drops which condense on the sides of the tube. How do you account for the reaction (R)? After most of the moisture is driven off, add water to the residue. Is it soluble? What is it? What is the industrial importance of this fact?

*b.* Add a few drops of ammonium hydroxide to a solution of magnesium chloride (R). Repeat the experiment, first diluting the magnesium chloride with an equal volume of a solution of ammonium chloride. Does a precipitate form? Since there are many magnesium ions present, what other ion must have largely disappeared? Can you account for this by mass action? To the solution add disodium phosphate. The precipitate has the formula  $\text{MgNH}_4\text{PO}_4$  (R).

**183. Weight of magnesium sulfate obtained from a known weight of magnesium oxide.** Weigh accurately a small evaporating dish; then introduce from 0.3 to 0.5 g. of magnesium

**P**

**F**

**L**

**M**

**N**

**O**

**P**

**Q**

**R**

**S**

**T**

**U**

**V**

**W**

**X**

**Y**

**Z**

oxide and accurately weigh the dish and contents. Add a little more than enough dilute sulfuric acid to dissolve the oxide (1 part acid to 3 parts water), and carefully evaporate to dryness, completing the operation under the hood. Moisten the powder with 1 or 2 drops of the acid and again evaporate, finally heating the product to a low red heat. Allow the crucible to cool, and weigh accurately the resulting magnesium sulfate. Compare your results with the theoretical results.

**184. Zinc.** *a.* Place a small piece of zinc on charcoal and heat it in the oxidizing-flame of the blowpipe (R). What is the color of the product while hot? Does its color change on cooling?

*b.* Try the solubility of zinc oxide in sodium hydroxide. Could a film of oxide remain on a piece of zinc in a solution of this reagent? If zinc is perfectly clean, how would you expect it to act with water? (See electromotive series.) Should zinc be soluble in a solution of sodium hydroxide? Try it.

*c.* Repeat § 182, *b*, substituting zinc sulfate for magnesium chloride.

**185. Combining weight of zinc.** Repeat § 183, substituting for the magnesium oxide about 1 g. of pure zinc, accurately weighed, and for the sulfuric acid, dilute nitric acid. What is the product first formed? What is obtained on heating to a low red heat? From the values obtained, calculate the combining weight of zinc referred to oxygen. How does this compare with the combining weight of zinc referred to hydrogen as unity as determined in § 85?

**186. Cadmium.** Obtain about 5 cc. of a solution of a salt of cadmium, and add to it a little dilute hydrochloric acid. Then pass hydrogen sulfide into the solution or add a solution of the reagent to it (R). What is the color of the precipitate? Is it soluble in dilute hydrochloric acid? Test its solubility in concentrated hydrochloric acid (?). For what is it used?



**187. Analytical reactions.** Pour into separate test tubes a solution of a compound of magnesium, zinc, and cadmium. Add a few drops of hydrochloric acid to each solution and then pass in hydrogen sulfide. Note the result (R). What would you infer as to the solubility of the sulfides of these metals in dilute acids? Add ammonium sulfide to separate solutions of compounds of magnesium and zinc. Repeat, adding an equal volume of ammonium chloride solution before adding the ammonium sulfide. Explain (R). How could you detect the three elements in the presence of each other?



## CHAPTER XXXI

### ALUMINIUM

**188. Aluminium and its hydroxide.** *a.* Dissolve a small piece of aluminium in hydrochloric acid. Where must the metal stand in reference to hydrogen in the electromotive series? Add sodium hydroxide, a drop at a time, until a precipitate is produced (R). Continue the addition with frequent stirring (R). When solution has been effected, add hydrochloric acid, a drop at a time (R). By what name would you designate a hydroxide with such properties?

*b.* Try the action of aluminium on boiling water. From the place of the metal in the electromotive series would you expect it to decompose water? Now try the action of the metal on a solution of sodium hydroxide (R). Does the fact that the hydroxide is soluble in alkalies suggest a reason, for the fact that, while the metal is not acted upon by water, it dissolves in alkalies? Polish the surface of a piece of aluminium and dip it into a solution of mercuric chloride. What becomes of the mercury (electromotive series)? The mercury keeps the hydroxide from sticking to the aluminium. Does water now attack the metal?

*c.* Try the action of a solution of ammonium hydroxide on a solution of aluminium chloride (R). Is the hydroxide dissolved by an excess of ammonium hydroxide?

**189. Aluminium salts.** *a.* To a solution of aluminium sulfate (or any other soluble salt of the metal) add a solution of sodium carbonate. What gas is evolved? What solid is precipitated? How could you prove it is not a carbonate? Repeat the experiment, using ammonium sulfide in place of sodium carbonate (R). Pass hydrogen sulfide into a solution





of a salt of aluminium. Does a precipitate form? How could you test for zinc and aluminium in the presence of each other? How test for magnesium and aluminium?

*b.* Obtain about 1 g. of some dry compound containing aluminium, and heat it on charcoal with the blowpipe (R). Moisten the residue with a drop or two of a solution of cobalt nitrate and heat it once more. Note the color of the residue. This constitutes a test for compounds containing aluminium.

*c.* Repeat the experiment, substituting a compound of zinc for that of aluminium. The green product is called Rinmann's green.

*d.* Mix a little dry alum and sodium bicarbonate and rub them together. Pour a little water on the mixture and note the result.

**190. Aluminium nitride.** Mix thoroughly 10 g. of fine aluminium powder with 1 g. of lampblack. Place the mixture in the form of a cone on a brick or iron plate. In the top of the cone introduce a piece of magnesium ribbon about 5 cm. long. Now light the magnesium ribbon. The combustion progresses safely and without explosion. When the mass is cooled, note the crystals of aluminium nitride mixed with crystals of aluminium oxide. Place some of the product in a test tube and cover it with a solution of caustic soda, heat gently, and note odor of gas evolved (?). What possible use does this suggest for aluminium?

**191. Double salts. *a. Alums.*** What is a double salt? What is an alum? Calculate the weight of aluminium sulfate (remember that it is a hydrated salt) and of ammonium sulfate required for the preparation of 25 g. of crystallized alum. Dissolve these separately in hot water so that the combined volume will be about 75 cc. Unite the hot solutions and set the product aside to crystallize. Can you make out the form of the crystals? Test the reaction to litmus paper of a solution of a few pure crystals.

*b. Carnallite.* What is the formula of carnallite? Calculate the weight of the individual salts necessary to make 25 g.



of crystallized carnallite. Which of these are hydrated? Weigh out the required amount of magnesium chloride and about one third more than the required amount of potassium chloride, mix the salts, and dissolve them in the least possible volume of hot water. Allow the solution to cool, decant the mother liquor, and wash the crystals with a very little cold water. See if you can detect both magnesium and potassium in the crystals. Is this a double salt or a complex salt?



## CHAPTER XXXII

### THE IRON FAMILY

**192. Reactions of the ferrous ion.** *a.* Place about 1 g. of iron in a beaker and cover the material with water. Iron by alcohol dissolves the fastest, but tacks or clean turnings will do. Add dilute hydrochloric acid, a small portion at a time, so as to keep up a brisk evolution of gas (R). Note the odor. It is chiefly due to phosphine; how do you account for the presence of this substance? Note the choking effect of the gas when breathed. Hold a nonluminous flame over the beaker for a moment. How do you account for the flashes of light? Before the iron has all dissolved, filter and at once make the following tests, using 1 or 2 cc. for each.

*b.* Try the action of a solution of ammonium sulfocyanate ( $\text{NH}_4\text{CNS}$ ). Pass hydrogen sulfide into a little of the solution (R). Test the action of ammonium sulfide (R). How do you account for the difference in the results of the last two experiments? Add sodium hydroxide to some of the solution (R). What is the color of the precipitate? What change occurs after the contents of the test tube have stood exposed to air (R)?

**193. Ferrous ammonium sulfate.** Dissolve 10 g. of iron in dilute sulfuric acid and filter from the insoluble residue (what is it?). What weight of ferrous sulfate should be in the filtrate? Dilute it to about 50 cc. What would be an equimolecular weight of ammonium sulfate? Weigh out this amount and add it to the hot solution of ferrous sulfate. Set the beaker aside for the crystallization of the salt. Filter off the crystals, wash them with a very little cold water, and spread them on filter paper to dry. What is the formula?



**194. Reactions of the ferric ion.** *a.* As in § 192, dissolve about 1 g. of iron in dilute hydrochloric acid and then treat the solution with 3 or 4 cc. of aqua regia. (What does this produce (R)?) Use portions of the resulting solution for the experiments below.

*b.* To one portion of the solution obtained in *a* add ammonium hydroxide in excess (R). To another portion add sodium hydroxide (R). Is the product soluble in an excess of the reagent? Test a third portion with a solution of ammonium sulfocyanate (R). To a fourth add sodium carbonate in excess. What is the precipitate (R)? To a fifth portion add ammonium sulfide in excess. Is the product ferric sulfide (R)? How can you tell? Dissolve some of the precipitate in hydrochloric acid. The milky residue is sulfur. Can you account for it?

*c.* Boil the remainder of the solution prepared in *a*, with the addition of hydrochloric acid, until the aqua regia has been destroyed. Then add about half a gram of zinc, warming the whole to maintain a brisk evolution of hydrogen. What change in color do you note? From time to time test small portions with ammonium sulfocyanate, adding more zinc if necessary. What inference do you draw?

*d.* If you dissolve a little iron in nitric acid, would you expect to obtain a ferric salt or a ferrous salt? Try it. Obtain a crystal of ferric nitrate, put it in a test tube, and add a little water. How do you explain the result? Since nitric acid is a strong acid, how would you describe ferric hydroxide?

**195. Ferric ammonium sulfate.** Dissolve 10 g. of crystals of ferrous sulfate in 20 cc. of water. Add to the solution the amount of sulfuric acid necessary to convert the salt to the ferric state; then add nitric acid, a drop at a time, until the color no longer changes. Evaporate (hood) to a sirupy mass and dissolve in a little hot water. Add to the resulting solution the amount of ammonium sulfate necessary to form ferric ammonium sulfate, and heat gently until the salt is dissolved. Set the resulting solution aside to crystallize.





Examine the form of the crystals. To what class of compounds does ferric ammonium sulfate belong? Write the equations for the reactions involved.

**196. Potassium ferrocyanide and potassium ferricyanide.** What are the formulas of these two salts? Does either of them give reactions for ferrous or for ferric ions? What reactions will show this? Try the action of a solution of potassium ferrocyanide on a ferrous salt and then on a ferric salt (R). Make a similar experiment, using a solution of potassium ferricyanide and trying its action on a ferric and on a ferrous salt (R). Tabulate your results.

**197. Cobalt and nickel.** Test separate solutions of a salt of cobalt and a salt of nickel with the borax bead; with a solution of sodium hydroxide; with ammonium sulfide. Note the results (R).

**198. Detection of iron, aluminium, calcium, and magnesium in the presence of each other.** *a.* To solutions of a salt of iron (ferric) and of aluminium, respectively, add sodium hydroxide, a drop at a time, until an excess has been added (R). Note that the aluminium compound at first precipitated redissolves on the further addition of sodium hydroxide. To the resulting solution add hydrochloric acid until the solution becomes acid, then make it slightly alkaline with ammonium hydroxide and heat to boiling. Note the result. Filter off the precipitate obtained by adding sodium hydroxide to the iron compound, dissolve a small portion of it in dilute hydrochloric acid, and add a drop of potassium sulfocyanate (R). How can you detect iron and aluminium compounds in the presence of each other?

*b.* Pour into separate test tubes about 2 cc. of solutions of separate compounds of each of the following metals: iron, aluminium, calcium, magnesium. Add to each an equal volume of ammonium chloride solution and then a few drops of ammonium hydroxide. Note the results. How can you separate iron and aluminium from calcium and magnesium?

*c.* To a solution of a salt of calcium and a solution of a salt of magnesium, respectively, add an equal volume of



ammonium chloride solution, then a few drops of ammonium carbonate solution (R). How would you verify the presence of magnesium?

*d.* Prepare an outline for the detection of compounds of iron, aluminium, calcium, and magnesium when mixed together in the same solution. Present the outline to the instructor for criticism and approval.

*e.* Dissolve about 1 g. of limestone in dilute hydrochloric acid, adding 2 or 3 drops of nitric acid to oxidize any iron to the ferric state. What does the effervescence indicate? What is the solution likely to contain? Filter off any residue (silica) and test the filtrate for the presence of iron, aluminium, calcium, and magnesium, according to your outline. If the amount of iron and aluminium present is small, do not attempt to separate them from each other.



## CHAPTER XXXIII

### COPPER; MERCURY; SILVER

**199. Cuprous chloride.** Powder 5 g. of cupric sulfate and place it in a large test tube or a small beaker. Add 20 cc. of concentrated hydrochloric acid and about 10 g. of copper, preferably in a form that has a large surface. What ions are now present? What action should metallic copper have on the  $\text{Cu}^{++}$  ion? Boil the mixture vigorously for about ten minutes (hood), and then pour it into a beaker containing at least 200 cc. of water. The white precipitate is cuprous chloride ( $\text{CuCl}$ ) (R). Decant the solution and wash the precipitate once with water. Boil a little of the cuprous chloride suspended in water (R). Treat a small portion with a solution of sodium hydroxide (R). Warm a third portion with aqua regia (R).

**200. Cuprous iodide.** To 2 or 3 cc. of a solution of cupric sulfate add about 1 cc. of a solution of potassium iodide (R). How do you account for the separation of iodine? How can you remove the latter from the insoluble cuprous iodide? Which is the more easily prepared, cuprous chloride or cuprous iodide?

**201. Reactions of cupric salts.** *a.* Recall the action of sulfuric and of nitric acid on copper (R). Dip a nail into a solution of cupric sulfate (R). What other metals would act like iron?

*b.* To a cold solution of cupric sulfate add one half its volume of a solution of sodium hydroxide (R). Heat the solution to boiling and account for the change in the color of the precipitate (R). Is cupric hydroxide soluble in sodium hydroxide?



c. Try the action of a solution of hydrogen sulfide on cupric sulfate (R); of a solution of ammonium sulfide (R). Add a drop of ammonium hydroxide to a dilute solution of cupric sulfate (R). Continue to add the ammonium hydroxide, drop by drop, until the precipitate dissolves. How does the color of the solution compare with that of the original cupric sulfate? Can there be any considerable number of cupric ions in the solution? Why?

d. To 5 cc. of a solution of cupric sulfate add 1 cc. of hydrochloric acid; then add an equal volume of a solution of hydrogen sulfide (R). Repeat with a solution of zinc sulfate. Try the action of ammonium sulfide upon zinc sulfate. How can you detect copper and zinc in the presence of each other?

e. From the results obtained in § 37 calculate the number of molecules of water of hydration present in cupric sulfate.

**202. Tetraminocuprisulfate ( $\text{Cu}(\text{NH}_4)_2\text{SO}_4 \cdot \text{H}_2\text{O}$ ).** Powder about 10 g. of cupric sulfate and dissolve it in 20 cc. of concentrated ammonium hydroxide and 10 cc. of water. While vigorously stirring the solution, add, drop by drop, about 25 cc. of alcohol. Collect the purple-blue crystalline precipitate on a filter. Can you prove that the compound contains copper? that it contains ammonia? that it is a sulfate?

**203. Analysis of brass.** To detect the presence of copper and zinc in brass, place 0.5 g. of brass in an evaporating-dish (hood) and dissolve it in as little nitric acid as possible. Note the color of the solution. What does it indicate? Evaporate the solution just to dryness, add 5 cc. of hydrochloric acid and 5 cc. of water, and warm gently. Transfer the clear liquid to a beaker, dilute to 100 cc. with hot water, and pass a slow current of hydrogen sulfide through the solution as long as a precipitate forms. What is the precipitate? Filter, and test the filtrate with hydrogen sulfide in order to be certain that the precipitation is complete. If no more precipitate forms, evaporate the filtrate to half its volume, add a few drops of nitric acid, and again heat to boiling. When the solution is cool, add ammonium hydroxide until it is alkaline, then warm gently and set aside for a few minutes. A slight precipitate





of ferric hydroxide ( $\text{Fe}(\text{OH})_3$ ) may form, due to traces of iron in the brass. Filter, and add ammonium sulfide to the clear filtrate. What is the composition of the precipitate?

**204. Mercuric compounds.** *a.* Note the physical properties of mercury. Place in a small beaker a globule of mercury as large as a grain of wheat, and add (hood) just enough concentrated nitric acid to dissolve it. Write the equation for the reaction on the supposition that mercuric nitrate is formed. Dilute the product with 10 cc. of water and place a copper cent in the solution. After a few minutes remove the coin and polish it with a piece of cloth. Account for the result.

*b.* For what purpose have we used mercuric oxide? Place 0.5 g. of it in a test tube and dissolve it in as little nitric acid as possible (R), then add water until the test tube is one fourth full. Divide the solution into three equal parts. To one part add a little hydrochloric acid (R); to a second part add ammonium hydroxide (is the product mercuric hydroxide? how can you prove it?); to the third part add a small piece of zinc or tin, and after a part of the metal has dissolved, add hydrochloric acid (R).

*c.* Prepare a little mercuric iodide by precipitation. What is its color? Collect it on a filter paper and allow it to dry. Scrape a little of the powder into a test tube and very carefully warm it at some distance above the flame. What change in color takes place? Is this change of color reversed on cooling?

**205. Mercurous compounds.** Put a small globule of mercury in a test tube and add a little dilute nitric acid, warming gently and taking care that some mercury remains undissolved. Divide the solution into three parts. To one part add hydrochloric acid or a soluble chloride (R), to a second add ammonium hydroxide (R), to the third add a little concentrated nitric acid (R). When action has ceased, add 1 or 2 cc. of hydrochloric acid (R). How can you distinguish between mercuric and mercurous salts?

**206. Compounds of silver.** *a.* Test the reaction of a solution of silver nitrate upon litmus paper. What would you conclude as to the strength of silver hydroxide as a base?



Place a drop of silver nitrate solution on a piece of cotton cloth and warm gently. Can you wash the stain away? What is it? Try ammonia water. Owing to the permanence of this stain, silver nitrate is used in making indelible ink.

*b.* To 5 cc. of a solution of silver nitrate add sodium hydroxide in excess (R). Wash the black precipitate repeatedly with hot water. Test its solubility in nitric acid (R); in ammonia water (R).

*c.* To 2 or 3 cc. of a solution of silver nitrate add hydrochloric acid (R). Is the chloride soluble in nitric acid? in ammonium hydroxide (R)? in sodium thiosulfate? Prepare sufficient silver sulfide to make note of its color and solubility in hydrochloric acid.

*d.* Prepare small amounts of the chloride, the bromide, and the iodide of silver (R). Expose to sunlight the test tubes containing the precipitates, and note any changes. For what are these compounds used? Prepare sufficient silver sulfide to note the color.

**207. Preparation of pure silver.** Place 2 or 3 g. of an alloy of copper and silver in a small beaker and add (hood) sufficient nitric acid to dissolve it. The solution may be hastened by applying a gentle heat. When the solution is complete, dilute the product with about 25 cc. of water. Account for the color of the liquid. Now add a solution of sodium chloride until a precipitate ceases to form. With stirring, the precipitate (what is it?) settles to the bottom of the beaker. Carefully decant the clear, supernatant liquid and test it for the presence of copper (§ 201, *c*). Wash the precipitate two or three times by pouring hot water over it and decanting. Finally, remove any remaining water by filtration. Mix the moist precipitate with an equal amount of sodium carbonate, transfer the mixture to a small cavity in a piece of charcoal, and heat it with a blowpipe. The silver salt is gradually reduced to metallic silver, which will fuse into a globule if sufficient heat is applied.



## CHAPTER XXXIV

### TIN AND LEAD

**208. Stannous compounds.** *a.* Dissolve about 0.5 g. of tin in hydrochloric acid (R). Why should this metal dissolve so much more slowly than zinc? Cool, dilute to 10 cc. with water, and use for the experiments below.

*b.* Place about 2 cc. of a solution of mercuric chloride in a test tube and add stannous chloride drop by drop (R). Does the color of the precipitate change with the continued addition of the reagent (R)?

*c.* To 1 or 2 cc. of the solution add a little aqua regia, boil until the acid is expelled, and repeat the experiment with mercuric chloride. Why does no precipitate form?

*d.* Pour a few drops into a solution of hydrogen sulfide (R).

*e.* Add a few drops of a solution of gold chloride to about 5 cc. of water. To 50 cc. of water add 1 or 2 drops of the solution of stannous chloride and 1 or 2 drops of a solution of sodium hydroxide. Add a little of this solution, drop by drop, to the solution of gold chloride. A deep rose-purple color slowly develops, known as the purple of Cassius. It is due to finely divided (colloidal) gold (R).

**209. Stannic compounds.** *a.* Add a few drops of concentrated nitric acid to a small piece of tin, heating gently if necessary. What is the compound formed? Is it soluble in water? in hydrochloric acid?

*b.* Dissolve a small piece of tin in aqua regia. What compound is formed (R)? Pour a few drops of the solution into a solution of hydrogen sulfide (R). Treat a little of the solution with a few drops of sodium hydroxide (R). Is the precipitate soluble in excess of the reagent?



**210. Crystallized stannic sulfide.** Mix together intimately 5 g. of stannous sulfide, 2 g. of ammonium chloride, and 2.5 g. of sulfur, placing the mixture in a test tube. Clamp the tube *loosely* in a nearly horizontal position (hood) and carefully heat it with a wing-top burner held in the hand, rotating the tube by twirling the rim with the fingers, and moving the burner constantly. Regulate the heat so that fumes of ammonium chloride are steadily evolved. When nearly all of the fumes have escaped, allow the tube to cool, and tap the crystals out on a piece of paper. To what element is sulfur similar? Is the conversion of stannous sulfide into stannic sulfide a process of oxidation?

**211. Reactions of lead. a.** Note the physical properties of the metal. Heat a small piece on charcoal in the oxidizing-flame. Note the incrustation formed (R).

**b.** Place 1 g. of the metal in an evaporating-dish (hood) and add 20 cc. of water and 5 cc. of nitric acid. Support the dish on a wire gauze and heat *gently* until the metal is dissolved (R). Evaporate until the volume is reduced about one half. What is the composition of the white body which separates? Dilute to 100 cc. and filter, if necessary, to obtain a clear solution. Now test small portions of this with hydrogen sulfide, sulfuric acid, potassium chromate, and ammonium carbonate respectively (R). Note the color and composition of the precipitates. Add a few drops of hydrochloric acid to a test tube one fourth full of the solution (R). Heat this to boiling, and if the solution does not become clear, add just enough boiling water to dissolve the precipitate; then set it aside until cool and note the result. How can you distinguish between lead chloride and silver chloride? In the remainder of the solution formed by dissolving the lead in nitric acid, suspend a piece of zinc. Set aside for half an hour and note the result (R). Test the solution for the presence of zinc, giving the method used.

**212. Oxides of lead.** Give the names and formulas of the oxides of lead. Place about 1 g. of lead peroxide in each of two test tubes. To the one add a few drops of hydrochloric acid; to the other, a few drops of sulfuric acid. Heat each





gently and note the result (R). What other compound reacts with these acids in a similar way? Treat a little red lead with dilute nitric acid and heat gently. After the action ceases, dilute and filter. Test the filtrate for lead. Test the residue on the filter paper by heating with hydrochloric acid (R). Try the action of hydrochloric acid and nitric acid on litharge (R).

**213. The storage battery.** *a.* Prepare the apparatus illustrated in Fig. 55. The tube *A* is the large test tube that was used in the preparation of oxygen; *B* and *C* are strips of sheet lead rubbed bright with coarse sandpaper. These strips should be about 1 cm. in width, and should be bent at right angles near one end so as to rest on the rim of the test tube. In this bent end a small hole is made by driving a wire nail through the lead, and a copper wire is passed through the hole and twisted to a tight connection. When the lead plates are suspended in the tube *A*, they must be far enough apart to be separated by a glass tube *D*, so as to prevent short-circuiting. The whole cell may be slipped into a wide-mouthed bottle as a support. *E* and *F* are binding-posts, terminals of two storage cells connected in series.

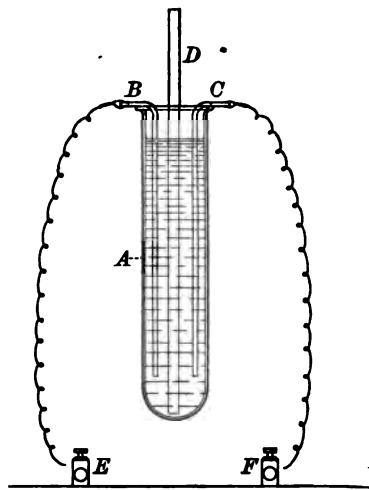


FIG. 55

*b.* As a preliminary experiment obtain 1 or 2 cc. of a solution of starch (side shelf) and dissolve in it a crystal of potassium iodide. Place a clean sheet of filter paper on a clean and dry spot on the table close to the binding-posts *E* and *F*, and on this pour a few drops of the solution just prepared. Attach two wires to the binding-posts and bring the two free ends upon the moist starch paper about 1 cm.



apart. Is there any change of color where the wires touch the paper? At which pole does it occur? How do you explain it (R)? Will this reaction serve to detect an electric current?

c. Prepare an electrolyte for the cell by mixing equal volumes of concentrated sulfuric acid and water, making enough of the solution to fill the tube *A* nearly full. When the solution has cooled, pour it into the tube *A* and bring the ends of the two wires connected with *B* and *C* upon the starch paper. Can you detect any current? Is your cell acting as a battery? Now connect the wires with the binding-posts. What changes in the cell do you note? Which plate turns brown? What is the brown substance? What is formed at the other plate? What gases are evolved? When one plate has become decidedly brown, disconnect the wires from *E* and *F* and bring the free ends upon the starch paper, assuring yourself that it is still moist. Is iodine set free now? What is the source of the current (R)?

**214. Analysis of solder.** What two metals are present in ordinary solder? Devise a method for detecting their presence in it and submit it to the instructor for approval. When your method has been approved, try it.



## CHAPTER XXXV

### MANGANESE AND CHROMIUM

**215. Salts of manganese.** To a solution of manganous chloride add a solution of hydrogen sulfide (R); a solution of ammonium sulfide (R). Try the action of sodium hydroxide and of ammonium hydroxide (R). Is the precipitate soluble in excess of the reagents? Does it undergo any change on exposure to the air (R)?

**216. Manganates and permanganates.** *a.* In a mortar grind 5 g. each of manganese dioxide and potassium hydroxide and 2.5 g. of potassium chlorate. Transfer the mixture to a small iron dish and heat to redness until the mass fuses and becomes deep green. The green material has the composition  $K_2MnO_4$  (R). When the mass has cooled, add cold water and dissolve as much of it as possible, decanting the solution from the residue.

*b.* The manganate is stable only in the presence of an excess of alkali. What would you argue as to the strength of manganic acid? In water it decomposes according to the equation



Add dilute nitric acid to the solution. What change in color do you note? The compound  $KMnO_4$  is deep purple in color. Does a precipitate form? What is it? Remembering that the reaction is reversible, what is the reason that nitric acid promotes the reaction from left to right? How would you promote the reaction from right to left? Try it.

*c.* In the presence of sulfuric acid, how does potassium permanganate act as an oxidizing agent (R)? What becomes



of the manganese? Are these compounds notably colored? Add a little acidified solution of permanganate to a solution of ferrous sulfate. How do you account for the disappearance of the color (R)? In a similar way try the action of permanganate upon oxalic acid and sulfurous acid (R). Pour a little hydrochloric acid upon two or three crystals of potassium permanganate (R).

**d.** Make a solution of potassium permanganate by adding 1 g. of the salt to 100 cc. of water, and fill a burette with the solution. Weigh accurately 0.5 g. of pure iron wire and dissolve it in an excess of dilute sulfuric acid. What is present in the solution? Slowly add the permanganate from the burette, constantly stirring the solution. When the color of a drop added fades rather slowly, add the solution more carefully, stopping the instant a permanent pink color is produced (R). The continuance of the color shows that all of the ferrous sulfate has been oxidized. From the weight of the iron oxidized, calculate the weight of permanganate used up. From the volume of the permanganate, calculate the quantity of the permanganate in a liter of the solution. Knowing the strength of the solution, could you determine the amount of iron present in a sample of unknown composition? This is a favorite method of analysis.

**217. Salts of chromium.** Try the effect of the following reagents upon a solution of a chromium salt, writing all the equations: ammonium sulfide, sodium carbonate, ammonium hydroxide. Does sodium hydroxide occasion a precipitate (R)? Is this soluble in excess of the reagent (R)? Try boiling the solution.

**218. Chromates and dichromates.** *a.* In an iron dish melt equal weights of potassium hydroxide and potassium nitrate (about 5 g. of each), and gradually stir into the mixture about 5 g. of finely powdered chrome iron ore or 3 g. of chromic oxide (R). The nitrate is used as an oxidizing agent. Allow the fused mass to cool, and extract the soluble portion with hot water. What is the color? To what is it due? To 3 or 4 cc. of the solution add nitric or sulfuric acid.





What change in color do you note (R)? Add potassium hydroxide to this solution. Is the color change reversed (R)? Write the equation for the transformation of a chromate to a dichromate as a reversible reaction. Why does the addition of acid throw it one way, while bases throw it the other?

*b.* To a solution of a chromate add a solution of a soluble salt of lead (R). Repeat, using a salt of barium (R). Repeat these experiments, using a solution of a dichromate instead of a solution of a chromate (R).

*c.* Pour 2 or 3 cc. of concentrated hydrochloric acid upon a few crystals of a chromate (R). Repeat, using potassium dichromate (R).

*d.* Place three or four crystals of potassium dichromate in a test tube and add 2 cc. of concentrated sulfuric acid. The deep-red crystals are chromic anhydride ( $\text{CrO}_3$ ) (R). Pour the mixture into cold water. Do the crystals dissolve? What do they form (R)?

## APPENDIX

### DATA FOR CALCULATION OF GAS VOLUMES

The relation between the volume of a gas under standard conditions,  $V_s$ , and its volume  $V$  when measured over water at a pressure  $P$ , an absolute temperature  $T$ , and an aqueous tension  $a$  is expressed by the equation

$$V_s = \frac{(P - a) \times V \times 273}{760 \times T}.$$

In comparing the pressure exerted by a column of water with that exerted by a column of mercury, it must be remembered that the density of mercury is 13.56.

### TENSION OF AQUEOUS VAPOR AT VARIOUS TEMPERATURES EXPRESSED IN MILLIMETERS OF MERCURY

TEMPERA- TURE	PRES- SURE	TEMPERA- TURE	PRES- SURE	TEMPERA- TURE	PRES- SURE
9° . . . . .	8.61	19° . . . . .	16.56	29° . . . . .	29.94
10° . . . . .	9.20	20° . . . . .	17.51	30° . . . . .	31.74
11° . . . . .	9.84	21° . . . . .	18.62	31° . . . . .	33.57
12° . . . . .	10.51	22° . . . . .	19.79	32° . . . . .	35.53
13° . . . . .	11.23	23° . . . . .	21.02	33° . . . . .	37.59
14° . . . . .	11.98	24° . . . . .	22.32	34° . . . . .	39.75
15° . . . . .	12.78	25° . . . . .	23.69	35° . . . . .	42.02
16° . . . . .	13.62	26° . . . . .	25.13	36° . . . . .	44.40
17° . . . . .	14.52	27° . . . . .	26.65	37° . . . . .	46.90
18° . . . . .	15.46	28° . . . . .	28.25	100° . . . . .	760.00

### WEIGHT IN GRAMS OF 1 LITER OF VARIOUS GASES UNDER STANDARD CONDITIONS, AND BOILING POINTS UNDER PRES- SURE OF 760 MILLIMETERS

NAME	WEIGHT OF 1 LITER	BOILING POINT	NAME	WEIGHT OF 1 LITER	BOILING POINT
Acetylene . . .	1.1621	-83.8°	Hydrogen chloride .	1.6398	-83.1°
Air . . . . .	1.2928		Hydrogen fluoride .	0.893	+19.4°
Ammonia . . .	0.7708	-33.5°	Hydrogen sulfide .	1.5392	-61.6°
Argon . . . . .	1.7809	-186.0°	Methane . . . . .	0.7168	-164.0°
Carbon dioxide .	1.9768	-78.2°	Nitric oxide . . .	1.3402	-153.0°
Carbon monoxide .	1.2504	-190.0°	Nitrogen . . . . .	1.2507	-195.7°
Chlorine . . . .	3.1674	-33.6°	Nitrous oxide . . .	1.9777	-89.8°
Helium . . . . .	0.1782	-268.7°	Oxygen . . . . .	1.4290	-183.0°
Hydrogen . . . .	0.08987	-252.7°	Sulfur dioxide . .	2.9266	-8.0°

## ELECTROCHEMICAL SERIES OF METALS

1. Potassium.	5. Zinc.	9. Hydrogen.	13. Mercury.
2. Sodium.	6. Iron.	10. Copper.	14. Silver.
3. Magnesium.	7. Tin.	11. Antimony.	15. Platinum.
4. Aluminium.	8. Lead.	12. Bismuth.	16. Gold.

## OUTFIT FOR EACH STUDENT LOCKER

### A. ARTICLES RETURNABLE

2 Bunsen burners.	2 beakers, 225-cc.
1 burner, wing-top.	2 beakers, 325-cc.
1 burette clamp.	1 bottle, narrow-mouthed, 1000-cc.
1 screw clamp.	1 bottle, wide-mouthed, 1000-cc.
1 set cork borers, Nos. 1-3.	4 bottles, wide-mouthed, 250-cc.
1 hemispherical iron dish, 75-mm.	2 bottles, wide-mouthed, 80-cc.
1 lead dish.	1 drying-tube, straight.
1 pair forceps.	1 flask, Erlenmeyer, 120-cc.
1 pinchcock.	1 flask, Florence, 120-cc.
1 deflagrating-spoon.	1 flask, Florence, 250-cc.
1 test-tube holder.	1 flask, Florence, 500-cc.
1 test-tube rack.	2 funnels, 85-mm.
1 mortar and pestle, 75-mm.	3 glass plates, 75-mm. $\times$ 75-mm.
1 piece platinum wire.	12 test tubes, 130-mm.
1 porcelain spatula, 130-mm.	2 test tubes, hard-glass, 150-mm.
1 casserole, 75-mm.	1 test tube, graduated, 30-cc.
1 porcelain crucible and lid, No. 00.	1 funnel tube.
1 porcelain dish, No. 0.	1 glass retort, 200 cc.
2 beakers, 60-cc.	1 U-tube, Marchand, 100-mm.
2 beakers, 100-cc.	1 watch glass, 78-mm.
2 beakers, 150-cc.	1 Sargent lock and 2 keys.

### B. ARTICLES NOT RETURNABLE

1 towel.	1 oz. of light-weight glass rod.
1 clay triangle.	1 cake soap.
1 test-tube brush.	3 ft. of rubber tubing, 4-mm., No. 22.
1 sponge.	6 ft. of rubber tubing, 6-mm., No. 22.
50 splints.	2 rubber stoppers, two-hole, No. 6.
1 box matches.	1 rubber stopper, one-hole, No. 5.
2 bottles litmus paper.	1 rubber stopper, one-hole, No. 4.
1 hard-glass tube, 500-mm. $\times$ 7-mm.	1 file, triangular.
1 hard-glass tube, 300-mm. $\times$ 10-mm.	1 file, round.
1 pkg. filter paper, 110-mm., No. 595.	1 wire gauze.
2 oz. of glass tubing, 6-mm.	

### APPARATUS TO BE AVAILABLE WHEN CALLED FOR

Burette, 50-cc. . . . .	1 for every 8 students
Cylinder, graduated, 150-cc. . . . .	1 for every 8 students
Cylinder, plain, 2500-cc. (for collecting gases in tubes) . . . . .	1 for every 8 students
Condenser, Liebig, 300-mm. . . . .	1 for every 4 students
Conductivity apparatus (see note to § 72, p. 148) . . . . .	1 for every 6 students
Gas tube, graduated, 50-cc. . . . .	1 for every 4 students
Gas tube, plain (bomb tube) . . . . .	1 for every 4 students
Magnet, horseshoe . . . . .	1 for every 10 students
Reading lens . . . . .	1 for every 10 students
Separatory funnel . . . . .	1 for every 4 students
Thermometer, ordinary centigrade . . . . .	1 for every 4 students
Thermometer graduated in tenths of a degree (10° — 30°) (see note to § 131, p. 150) . . . . .	1 for every 8 students
Victor Meyer apparatus . . . . .	2 or 3 sets

### APPARATUS PROVIDED FOR GENERAL USE

Balance and weights (see note to § 7, p. 148).

Barometer, mercurial.

Blast lamp.

Pneumatic trough (see note to § 12, p. 148). If locker space permits, the pneumatic trough should form a part of each outfit.

Ring stands. If movable ring stands are used, one should be in each outfit. It is better to provide the laboratory with permanent ring stands on each desk. These are easily made by screwing an iron rod into a flat disk that in turn is screwed to the desk top. Local workmen can make them.

### LIST OF CHEMICALS FOR A CLASS OF TEN STUDENTS

In the following list will be found an estimate of the actual quantities of reagents required for a class of ten students. In many cases these quantities are less than the smallest commercial package, and no supply house would want to furnish such small quantities. In the second column will be found the smallest commercial package that will be sufficient, the figures having been supplied through the kindness of the Kauffman-Lattimer Co. Columbus, Ohio. It will be noticed that in ordering for classes of large size it will not in all cases be necessary to multiply the number of commercial packages by the number of pupils in the class.

	ACTUAL QUANTITIES IN GRAMS	COMMERCIAL PACKAGES
Acid, acetic (36 per cent) . . . . .	110	$\frac{1}{4}$ lb.
Acid, formic (90 per cent) . . . . .	500	1 lb.
Acid, hydrochloric (sp. gr. 1.19) . . . . .	2000	6 lb.
Acid, nitric (sp. gr. 1.42) . . . . .	300	7 lb.
Acid, oxalic . . . . .	80	$\frac{1}{4}$ lb.
Acid, pyrogalllic . . . . .	40	1 oz.
Acid, sulfuric (sp. gr. 1.84) . . . . .	2000	9 lb.
Agar . . . . .	10	1 oz.
Alcohol . . . . .	500	1 qt.
Aluminium chloride . . . . .	20	1 oz.
Aluminium foil . . . . .	10	1 oz.
Aluminium sulfate . . . . .	30	$\frac{1}{4}$ lb.
Ammonium hydroxide (sp. gr. 0.90) . . . . .	1000	4 lb.
Ammonium carbonate . . . . .	100	$\frac{1}{4}$ lb.
Ammonium chloride . . . . .	500	1 lb.
Ammonium molybdate . . . . .	5	1 oz.
Ammonium nitrate . . . . .	80	$\frac{1}{4}$ lb.
Ammonium oxalate . . . . .	20	1 oz.
Ammonium sulfate . . . . .	100	$\frac{1}{4}$ lb.
Ammonium sulfocyanate . . . . .	10	1 oz.
Antimony . . . . .	50	2 oz.
Antimony oxide . . . . .	10	1 oz.
Antimony sulfate . . . . .	10	1 oz.
Arsenic . . . . .	10	1 oz.
Arsenic trioxide . . . . .	10	$\frac{1}{4}$ lb.
Barium chloride . . . . .	30	$\frac{1}{4}$ lb.
Barium hydrate . . . . .	10	1 oz.
Barium nitrate . . . . .	30	$\frac{1}{4}$ lb.
Barium peroxide . . . . .	50	2 oz.
Benzene . . . . .	500	1 pt.
Bismuth . . . . .	40	2 oz.
Bismuth chloride . . . . .	10	$\frac{1}{4}$ oz.
Bismuth nitrate . . . . .	30	1 oz.
Bone black . . . . .	30	$\frac{1}{4}$ lb.
Borax . . . . .	100	$\frac{1}{4}$ lb.
Brass turnings . . . . .	10	1 oz.
Bromine . . . . .	40	2 oz.
Cadmium sulfate . . . . .	25	1 oz.
Calcium carbide . . . . .	10	$\frac{1}{4}$ lb.
Calcium chloride . . . . .	500	1 lb.
Calcium fluoride (fluorite) . . . . .	40	$\frac{1}{4}$ lb.
Calcium nitrate . . . . .	20	1 oz.
Carbon disulfide . . . . .	80	$\frac{1}{4}$ lb.
Charcoal, powdered . . . . .	40	$\frac{1}{4}$ lb.

	ACTUAL QUANTITIES IN GRAMS	COMMERCIAL PACKAGES
Charcoal, sticks . . . . .		1 doz.
Chloroform . . . . .	100	$\frac{1}{2}$ lb.
Chromic oxide . . . . .	30	1 oz.
Cobalt nitrate . . . . .	30	1 oz.
Cochineal . . . . .		1 oz.
Copper foil . . . . .	50	2 oz.
Copper powder . . . . .	170	$\frac{1}{2}$ lb.
Copper turnings . . . . .	250	$\frac{1}{2}$ lb.
Cotton . . . . .		1 oz.
Cottonseed oil . . . . .	50	$\frac{1}{2}$ lb.
Cupric bromide . . . . .	15	$\frac{1}{2}$ oz.
Cupric chloride . . . . .	10	1 oz.
Cupric nitrate . . . . .	30	1 oz.
Cupric oxide . . . . .	140	$\frac{1}{2}$ lb.
Cupric sulfate . . . . .	250	$\frac{1}{2}$ lb.
Cuprous oxide . . . . .	30	1 oz.
Ferric chloride . . . . .	20	$\frac{1}{2}$ lb.
Ferric oxide . . . . .	10	1 oz.
Ferrous sulfate . . . . .	120	$\frac{1}{2}$ lb.
Ferrous sulfide . . . . .	250	1 lb.
Glucose . . . . .	1000	2 lb.
Hydrogen peroxide . . . . .	50	$\frac{1}{2}$ lb.
Iodine . . . . .	15	$\frac{1}{2}$ oz.
Iron (by alcohol) . . . . .	170	$\frac{1}{2}$ lb.
Lead acetate . . . . .	30	$\frac{1}{2}$ lb.
Lead nitrate . . . . .	30	$\frac{1}{2}$ lb.
Lead oxide (litharge) . . . . .	30	$\frac{1}{2}$ lb.
Lead peroxide . . . . .	30	1 oz.
Lime . . . . .	1000	2 lb.
Limestone . . . . .	60	1 lb.
Litmus cubes . . . . .	3	1 oz.
Magnesite . . . . .	50	$\frac{1}{2}$ lb.
Magnesium chloride . . . . .	180	$\frac{1}{2}$ lb.
Magnesium oxide . . . . .	10	2 oz.
Magnesium powder . . . . .	30	1 oz.
Magnesium ribbon . . . . .	15	1 oz.
Magnesium sulfate . . . . .	50	1 lb.
Manganese chloride . . . . .	10	1 oz.
Manganese dioxide (pyrolusite) . . . . .	200	$\frac{1}{2}$ lb.
Marble . . . . .	200	1 lb.
Mercuric chloride . . . . .	35	1 oz.
Mercuric oxide . . . . .	10	1 oz.
Mercuric sulfate . . . . .	10	1 oz.
Mercury . . . . .	1000	2 lb.

	ACTUAL QUANTITIES IN GRAMS	COMMERCIAL PACKAGES
Nickel nitrate . . . . .	20	1 oz.
Paraffin . . . . .	100	1 lb.
Phenolphthalein . . . . .		$\frac{1}{2}$ oz.
Phosphorus (red) . . . . .	50	2 oz.
Phosphorus (white) . . . . .	50	2 oz.
Phosphorus pentoxide . . . . .	10	1 oz.
Phosphorus trichloride . . . . .	15	1 oz.
Potassium alum . . . . .	30	1 lb.
Potassium bromide . . . . .	30	1 oz.
Potassium carbonate . . . . .	20	2 oz.
Potassium chlorate . . . . .	225	$\frac{1}{2}$ lb.
Potassium chloride . . . . .	225	$\frac{1}{2}$ lb.
Potassium dichromate . . . . .	100	$\frac{1}{2}$ lb.
Potassium ferricyanide . . . . .	10	1 oz.
Potassium ferrocyanide . . . . .	10	1 oz.
Potassium hydroxide . . . . .	250	$\frac{1}{2}$ lb.
Potassium iodide . . . . .	35	1 oz.
Potassium nitrate . . . . .	280	$\frac{1}{2}$ lb.
Potassium perchlorate . . . . .	20	1 oz.
Potassium permanganate . . . . .	475	1 lb.
Potassium sulfate . . . . .	10	$\frac{1}{2}$ lb.
Rochelle salts . . . . .	200	$\frac{1}{2}$ lb.
Silver nitrate . . . . .	30	1 oz.
Soda lime . . . . .	200	$\frac{1}{2}$ lb.
Sodium (metal) . . . . .	30	1 oz.
Sodium acetate . . . . .	270	$\frac{1}{2}$ lb.
Sodium ammonium phosphate . . . . .	20	1 oz.
Sodium bicarbonate . . . . .	30	1 lb.
Sodium bromide . . . . .	20	1 oz.
Sodium carbonate . . . . .	200	1 lb.
Sodium hydroxide . . . . .	400	1 lb.
Sodium iodide . . . . .	30	1 oz.
Sodium nitrate . . . . .	250	$\frac{1}{2}$ lb.
Sodium nitrite . . . . .	60	2 oz.
Sodium peroxide . . . . .	50	2 oz.
Sodium phosphate, disodium . . . . .	30	$\frac{1}{2}$ lb.
Sodium phosphate, trisodium . . . . .	10	1 oz.
Sodium sulfate . . . . .	300	1 lb.
Sodium sulfite . . . . .	130	1 lb.
Sodium thiosulfate . . . . .	30	1 lb.
Stannous sulfide . . . . .	50	2 oz.
Sulfur, powdered . . . . .	100	1 lb.
Sulfur roll . . . . .	100	1 lb.
Tartar emetic . . . . .	10	1 oz.

	ACTUAL QUANTITIES IN GRAMS	COMMERCIAL PACKAGES
Tin, mossy . . . . .	250	$\frac{1}{2}$ lb.
Zinc, granulated . . . . .	170	$\frac{1}{2}$ lb.
Zinc, mossy . . . . .	500	1 lb.
Zinc oxide . . . . .	10	$\frac{1}{2}$ lb.
Zinc sulfate . . . . .	40	1 lb.

#### MISCELLANEOUS SUPPLIES

Candles, beeswax (to be cut into pieces) . . . . .	2
Coal, powdered . . . . .	1 lb.
Coal oil . . . . .	1 qt.
Corks, Nos. 3, 6, 8, 10, 22, 24 . . . . .	2 doz. each
Emery paper, No. 0 . . . . .	2 sheets
Glazed paper . . . . .	6 sheets
Iron nails or tacks . . . . .	2 lb.
Iron wire (picture-frame) . . . . .	1 box
Labels, Dennison's, No. 219 . . . . .	2 boxes
Lead strips (see note to § 213, p. 150) . . . . .	10
Olive oil (for burns) . . . . .	1 pt.
Sand . . . . .	2 lb.
Sandpaper, No. 0 . . . . .	4 sheets
Sawdust (preferably hardwood) . . . . .	1 lb.
Starch . . . . .	$\frac{1}{2}$ lb.
Sugar . . . . .	1 lb.
Yeast . . . . .	1 cake

#### LABORATORY SOLUTIONS

Each working desk should be supplied with the common acids and alkalis, both concentrated and dilute. In preparing the dilute reagents, concentrated sulfuric acid should be diluted in the ratio 1 : 4; concentrated hydrochloric acid, nitric acid, and ammonia, in the ratio 1 : 3.

In preparing other solutions for the side shelf it is customary in most laboratories to make 10 per cent solutions, though it is much more satisfactory to make the solutions approximately normal in anhydrous salt. In the case of sparingly soluble salts, saturated solutions are used. With expensive reagents, such as silver nitrate or potassium iodide, a concentration of about 20 g. to the liter is sufficient for most uses.

It is convenient to have the bottles on the side shelf arranged on the principle of a wash bottle, a rubber tube being attached to



§ 131. The most convenient thermometer for use in this section is the type sold for use in constant-temperature thermostats. They are of small dimensions, having a scale reading from about 10° to 30°, and are graduated in tenths of a degree.

§ 213. The strips of lead used in this section should be cut from thin sheet lead such as can be had in any good tinner's shop. They should be about 1 cm. wide and 15 cm. long.

#### NATURAL GAS IN A BLAST LAMP

It is hard to make use of natural gas in a blast lamp, because the amount of air required for combustion cools the flame below the kindling temperature, and the flame is blown out. This may be remedied by diluting the gas with air before it reaches the burner. The easiest way to accomplish this is to make a by-pass tube leading from the hose supplying compressed air to the one supplying gas. The connection can be made by inserting a T-tube in each hose and connecting the two free ends with rubber tubing. A pinch clamp should be placed on this tubing to regulate the air admitted. See *Journal of Industrial and Engineering Chemistry*, VII, 46.





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# LIST OF THE AND

Aluminium . . .	Al				
Antimony . . .	Sb				
Argon . . . . .	A				
Arsenic . . . . .	As				
Barium . . . . .	Ba				
Bismuth . . . . .	Bi				
Boron . . . . .	B				
Bromine . . . . .	Br				
Cadmium . . . . .	Cd				
Cæsium . . . . .	Cs				
Calcium . . . . .	Ca				
Carbon . . . . .	C				
Cerium . . . . .	Ce				
Chlorine . . . . .	Cl				
Chromium . . . . .	Cr				
Cobalt . . . . .	Co				
Columbium . . . . .	Cb				
Copper . . . . .	Cu				
Dysprosium . . . . .	Dy				
Erbium . . . . .	Er				
Europium . . . . .	Eu				
Fluorine . . . . .	F				
Gadolinium . . . . .	Gd				
Gallium . . . . .	Ga				
Germanium . . . . .	Ge				
Glucinum . . . . .	Gl				
Gold . . . . .	Au				
Helium . . . . .	He				
Holmium . . . . .	Ho	163.5	Thallium . . . . .	Tl	204.0
Hydrogen . . . . .	H	1.008	Thorium . . . . .	Th	232.15
Indium . . . . .	In	114.8	Thulium . . . . .	Tm	168.5
Iodine . . . . .	I	126.92	Tin . . . . .	Sn	118.7
Iridium . . . . .	Ir	193.1	Titanium . . . . .	Ti	48.1
Iron . . . . .	Fe	55.84	Tungsten . . . . .	W	184.0
Krypton . . . . .	Kr	82.92	Uranium . . . . .	U	238.2
Lanthanum . . . . .	La	139.0	Vanadium . . . . .	V	51.0
Lead . . . . .	Pb	207.2	Xenon . . . . .	Xe	130.2
Lithium . . . . .	Li	6.94	Ytterbium . . . . .	Yb	173.5
Lutecium . . . . .	Lu	175.0	Yttrium . . . . .	Yt	89.3
Magnesium . . . . .	Mg	24.32	Zinc . . . . .	Zn	65.37
Manganese . . . . .	Mn	54.93	Zirconium . . . . .	Zr	90.6
Mercury . . . . .	Hg	200.6			

